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ORIGINAL PAPER

Efficient, green and regioselective synthesis of 1,4,5-trisubstituted-1,2,3-triazoles in ionic liquid [bmim]BF_4 and in task-specific basic ionic liquid [bmim]OH

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Abstract Convenient and environmentally benign procedures have been reported for the synthesis of 1,4,5-trisubstituted-1,2,3-triazoles by the reaction of aryl azides with active methylene compounds in ionic liquid [bmim]BF_4 in the presence of L-proline as a catalyst and also in task-specific basic ionic liquid [bmim]OH. The methodologies defined herein avoid the severe conditions as posed by earlier existing methods and proved to be efficient in terms of good yields, operational simplicity, easy workup and short reaction time.

Keywords 1,2,3-Triazoles · [bmim]BF_4 · [bmim]OH · Ionic liquids · Regioselective · L-Proline

Introduction

1,2,3-Triazoles are an important class of compounds because of their wide coverage of biological properties including antiviral [1], antimicrobial [2], anti-HIV [3, 4], anticonvulsants [5] and anti-allergic [6]. In addition, compounds having 1,2,3-triazole group have found industrial applications as dyes, corrosion inhibitors, sensors and photo-stabilizers [7]. The most popular method for the construction of 1,2,3-triazole framework, is the 1,3-dipolar cycloaddition reaction of azides with alkynes or enolizable compounds [8–10]. Copper (I)-catalyzed 1,3-dipolar cycloaddition of azides and alkynes is often used to prepare 1,5-disubstituted 1,2,3-triazoles [11–21]. However, the presence of the copper may induce degradation of viruses or oligonucleotide strands in biological systems [22, 23]. Copper ions are also potentially toxic for living organisms. Base catalyzed reactions of aryl azides with active methylene compounds also represent a powerful, though less developed, approach to a variety of monocyclic and bicyclic 1,2,3-triazole derivatives with different substitution patterns in positions 4 and 5 of the ring. The reported methods for 1,3-cycloaddition of aryl azides to active methylene compounds involve long reaction time [24], use of volatile catalysts [25] and toxic organic solvents such as DMSO and 1,4-dioxane [24, 26].

Room temperature ionic liquids have shown great promise as attractive alternatives to conventional volatile and toxic organic solvents [27, 28] owing to their negligible vapour pressure, recyclability, solvophobic properties, and ability to promote association of reactants in solvent cavity during activation process. Therefore, room temperature ionic liquids have received much attention as an alternative reaction media for catalytic processes and also as task-specific ionic liquids [29, 30]. In view of the emerging importance of ionic liquids as novel reaction media and the need for advent of novel methods that are devoid of listed deficiencies, we wish to report the use of ionic liquids in the synthesis of 1,4,5-trisubstituted-1,2,3-triazoles.

Experimental

Structures of all of the compounds were identified by their spectral data. Silica gel 60 F_{254} (precoated aluminium plates) from Merck was used to monitor reaction progress. Melting points were determined on a melting point

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apparatus and were uncorrected. IR (KBr) spectra were recorded on Perkin Elmer FTIR spectrophotometer and the values were expressed as ν_{max} cm^{-1}. Mass spectral data were recorded on a Waters Micromass Spectrometer running under Mass Lynex version 4.0 software and equipped with an ESI source. The NMR (^1H and ^{13}C) spectra were recorded on Jeol JNM ECX-400P at 400 and 100 MHz, respectively. The chemical shift values are recorded on δ scale and the coupling constants (J) are in Hertz. Different aryl azides were prepared from corresponding aryl amines by reported procedure [31].

General procedure for the synthesis of 1,4,5-trisubstituted-1,2,3-triazoles (1a–1n)

Method A

A mixture of aryl azide (1.0 mmol), acetylacetone or ethyl acetoacetate or methyl acetoacetate (1.0 mmol) and L-Proline (10 mol%) was placed in a 50 mL round-bottomed flask containing 1.30 mL of ionic liquid, [bmim]BF_4. The mixture was stirred at 80 °C for appropriate time as mentioned in Table 1. After completion of reaction as monitored by TLC using ethyl acetate:petroleum ether (60:40, v/v) as eluent, the reaction mixture was allowed to cool to room temperature and was quenched with water (10 mL). The precipitate formed was collected by filtration at pump, washed with water and dried to yield pure 1,4,5-trisubstituted-1,2,3-triazoles (**1a**–**1n**).

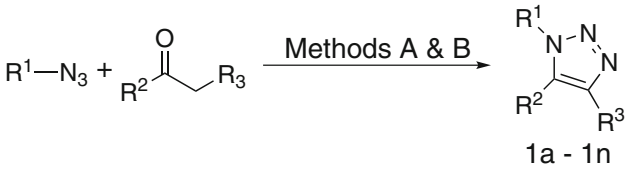
Method B

A mixture of aryl azide (1.0 mmol), acetylacetone or ethyl acetoacetate or methyl acetoacetate (1.0 mmol) was placed in a 50 mL round-bottomed flask containing task-specific basic ionic liquid [bmim]OH (10.0 mmol). The mixture was stirred at 80 °C for appropriate time as mentioned in Table 1. After completion of the reaction as monitored by TLC using ethyl acetate:petroleum ether (60:40, v/v) as eluent, the reaction mixture was allowed to cool to room temperature and reaction was quenched with water (10 mL). The precipitate formed was collected by filtration at pump, washed with water and dried to yield pure 1,4,5-trisubstituted-1,2,3-triazoles (**1a**–**1n**).

X-ray structure determination

X-ray intensity data for compound **1k** was collected on Oxford Diffraction Xcalibur CCD diffractometer with

Table 1 Synthesis of various 1,4,5-trisubstituted-1,2,3-triazoles



Entry	R^1	R^2	R^3	Product	Method A		Method B			Lit.Mp (°C)
					Time (min)	Yield (%)	Time (min)	Yield (%)	Mp (°C)	
1	4-$NO_2C_6H_4$	CH_3	$COCH_3$	**1a**	45	87	35	93	145–147	148 [32]
2	4-ClC_6H_4	CH_3	$COCH_3$	**1b**	85	75	65	87	116–118	119 [32]
3	4-BrC_6H_4	CH_3	$COCH_3$	**1c**	60	79	50	89	120–122	120 [32]
4	4-MeC_6H_4	CH_3	$COCH_3$	**1d**	125	74	80	82	118–120	120 [32]
5	4-$MeOC_6H_4$	CH_3	$COCH_3$	**1e**	140	75	95	79	120–121	120 [32]
6	C_6H_5	CH_3	$COCH_3$	**1f**	120	70	75	84	108–110	108 [32]
7	4-FC_6H_4	CH_3	$COCH_3$	**1g**	65	85	50	95	80–82[a]	–
8	4-F,3-ClC_6H_3	CH_3	$COCH_3$	**1h**	55	87	45	90	95–97	–
9	4-$NO_2C_6H_4$	CH_3	$COOCH_2CH_3$	**1i**	60	80	40	94	124–125	–
10	4-ClC_6H_4	CH_3	$COOCH_2CH_3$	**1j**	95	79	65	85	90–92[a]	–
11	4-BrC_6H_4	CH_3	$COOCH_2CH_3$	**1k**	75	82	60	87	164–165	–
12	4-F,3-ClC_6H_3	CH_3	$COOCH_2CH_3$	**1l**	70	84	55	89	55–57	–
13	4-$NO_2C_6H_4$	CH_3	$COOCH_3$	**1m**	50	83	35	90	155–156	154 [33]
14	4-BrC_6H_4	CH_3	$COOCH_3$	**1n**	70	79	55	85	204–206	200 [33]

[a] Compound known but melting point reported for first time

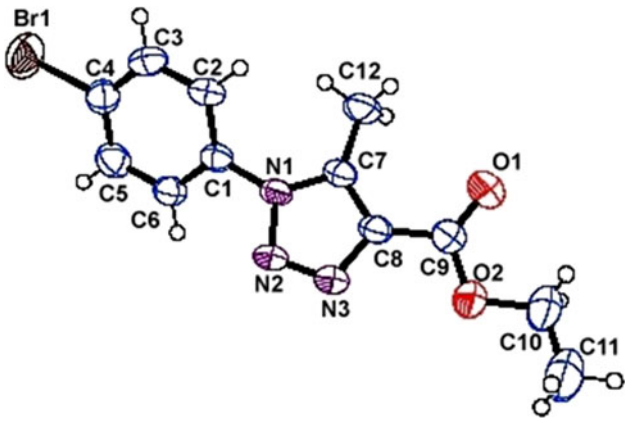


Fig. 1 ORTEP diagram showing X-ray structure of compound **1k** drawn with 50 % ellipsoid probability

graphite monochromatic Mo Kα radiation ($\lambda = 0.71073$ Å) at temperature 298 K. Computations were carried out using WinGX-32 graphical user interface. The structure was solved by direct methods using *SIR97*. Data were refined and extended using *SHELX-97* software. Non-hydrogen atoms were refined anisotropically. Carbon-bound hydrogen atoms were included in idealized positions and refined using a riding model. Crystallographic data (excluding structure factors) for the structure have been deposited with the Cambridge Crystallographic Data Center with CCDC No. 884194. These data can be obtained free of charge from the CCDC via http://www.ccdc.cam.ac.uk/data_request/cif. The single crystal image is given in Fig. 1.

Crystallographic data

Chemical formula: $C_{12}H_{12}BrN_3O_2$, crystal color: colorless, crystal dimensions: 0.58 mm (max), 0.15 mm (mid), 0.07 mm (min), crystal system: monoclinic, space group: *P* 1 21/c 1, unit cell parameters: $a = 13.7062(9)$, $b = 7.7246(5)$, $c = 12.5297(9)$, number of formula units in the unit cell (Z) = 4, absorption coefficient [μ (mm^{-1})] = 3.165, cell measurement theta = 3.0371 (min), 29.1657 (max), goodness of fit (S) = 1.044, final R values: R (reflections) = 0.0429 (1,705), wR2 (reflections) = 0.1170 (2,285).

Spectral data of new compounds

1-(1-(3-Chloro-4-fluorophenyl)-5-methyl-1H-1,2,3-triazol-4-yl)ethanone ***1h***

IR (KBr, cm^{-1}): $\nu_{max} = 3{,}074$, 1,685, 1,559; ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 7.45 (d, 1H, $J = 6.6$ Hz, Ar–H), 7.24 (d, 2H, $J = 6.6$ Hz Ar–H), 2.62 (s, 3H, $COCH_3$), 2.47 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.2, 160.3, 143.5, 137.4, 127.8, 125.2, 125.1, 117.7, 117.5, 27.8, 10.0; HRMS (ESI): $m/z = 254.4649$ [M^++1].

Ethyl 1-(4-nitrophenyl)-5-methyl–1H-1,2,3-triazole-4-carboxylate ***1i***

IR (KBr, cm^{-1}): $\nu_{max} = 3{,}079$, 1,720, 1,560, 1,532; ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 8.44 (d, 2H, $J = 8.76$ Hz, Ar–H), 7.71 (d, $J = 8.8$ Hz, 2H, Ar–H), 4.42–4.45 (q, $J = 7.32$ Hz, 2H, OCH_2), 2.66 (s, 3H, CH_3), 1.40–1.44 (t, $J = 7.32$ Hz, 3H, CH_2 CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.3, 148.1, 140.2, 138.8, 137.4, 125.8, 125.1, 61.3, 14.3, 10.1; HRMS (ESI): $m/z = 277.4718$ [M^++1].

Ethyl 1-(4-bromophenyl)-5-methyl-1H-1,2,3-triazole-4-carboxylate ***1k***

IR (KBr, cm^{-1}): $\nu_{max} = 3{,}096$, 1,718, 1,586, 1,563; ^{1}H NMR (400 MHz, DMSO) δ_H: 7.85 (d, 2H, $J = 8.8$ Hz, Ar–H), 7.60 (d, $J = 8.76$ Hz, 2H, Ar–H), 4.31–4.36 (q, $J = 7.32$ Hz, 2H, OCH_2), 2.50 (s, 3H, CH_3), 1.29–1.33 (t, $J = 7.36$ Hz, 3H, CH_2 CH_3); ^{13}C NMR (100 MHz, DMSO) δ 161.4, 142.1, 139.6, 134.3, 132.7, 127.5, 123.4, 60.5, 14.1, 9.7; HRMS (ESI): $m/z = 310.3614$ [M^++1].

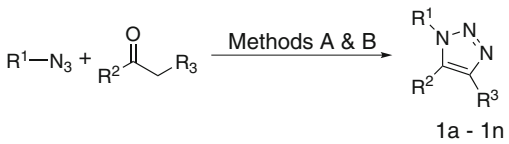
Ethyl 1-(3-chloro-4-fluorophenyl)-5-methyl-1H-1,2,3-triazole-4-carboxylate ***1l***

IR (KBr, cm^{-1}): $\nu_{max} = 3{,}073$, 1,720, 1,570, 1,509; ^{1}H NMR (400 MHz, CDCl3) δ_H: 7.55 (d, 1H, $J = 6.6$ Hz, Ar–H), 7.33 (d, 2H, $J = 6.6$ Hz Ar–H), 4.41–4.46 (q, $J = 7.32$ Hz, 2H, OCH_2), 2.57 (s, 3H, CH_3), 1.40–1.43 (t, $J = 7.32$ Hz, 3H, CH_2 CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.4, 157.5, 138.9, 136.8, 127.9, 125.3, 125.2, 117.7, 117.5, 61.2, 14.3, 9.9; HRMS (ESI): $m/z = 284.4142$ [M^++1].

Results and discussion

We present herein an efficient and environmentally benign protocol for the synthesis of 1,4,5-trisubstituted-1,2,3-triazoles by the reaction of various aryl azides with active methylene compounds in ionic liquid [bmim]BF_4 in the presence of L-proline as a catalyst and also in task-specific basic ionic liquid [bmim]OH which acts as a reaction medium as well as catalyst (Scheme 1).

To achieve optimum reaction conditions, we investigated the reaction of 4-nitrophenyl azide (1.0 mmol) and acetylacetone (1.0 mmol) as model substrates in ionic liquid [bmim]BF_4 in the presence of basic catalysts such as Et_3NH, K_2CO_3, and L-proline. The best results were obtained when the reaction was carried out in [bmim]BF_4



Scheme 1 Synthesis of 1,4,5-trisubstituted-1,2,3-triazoles

as reaction medium in the presence of catalytic amounts of L-proline (10 mol%) at 80 °C. The reaction underwent completion in 45 min yielding 87 % of 1-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)ethanone (**1a**) after a simple work up (entry 1, "Method A", Table 1). The reaction in [bmim]BF_4 at 80 °C in the absence of catalyst resulted in incomplete reaction even after 12 h. The reactions using catalytic amounts of Et_3NH (10 mol%), K_2CO_3 (10 mol%) and L-proline (5 mol%) in [bmim]BF_4 at 80 °C required long reaction times and gave inferior yields. Further, reaction in the presence of 15 mol% of L-proline as a catalyst did not affect the reaction time and yield significantly. Therefore, L-proline (10 mol%) in ionic liquid [bmim]BF_4 at 80 °C was chosen as optimum system for this protocol. Subsequently, reactions of other substituted aromatic azides were carried out under these conditions. The reactions proceeded smoothly for different aryl azides to produce the corresponding 1,4,5-trisubstituted-1,2,3-triazoles (**1b**–**1h**) in high yields.

The protocol was further extended by exploring the 1,3-cycloaddition of aryl azides with other active methylene compounds, e.g. ethyl acetoacetate and methyl acetoacetate. The reaction of 4-nitrophenyl azide (1.0 mmol) with ethyl acetoacetate (1.0 mmol) in ionic liquid [bmim]BF_4 in the presence of 10 mol% of L-proline at 80 °C yielded 80 % of ethyl 5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazole-4-carboxylate (**1i**). Similarly, other azides also gave corresponding triazoles (**1j**–**1l**) in high yields. Also reactions of 4-nitrophenyl azide (1.0 mmol) and 4-bromophenyl azide (1.0 mmol) with methyl acetoacetate (1.0 mmol) under similar reaction conditions yielded 83 and 79 % of methyl 5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazole-4-carboxylate (**1m**) and methyl 5-methyl-1-(4-bromophenyl)-1*H*-1,2,3-triazole-4-carboxylate (**1n**), respectively. These results have been summarized in Table 1 ("Method A").

We further decided to explore this reaction using task-specific basic ionic liquid [bmim]OH as a reaction medium as well as the catalyst. Therefore, reaction of 4-nitrophenyl azide (1.0 mmol) and acetylacetone (1.0 mmol) was attempted in [bmim]OH without the aid of any catalyst at 80 °C. TLC analyses showed the reaction to be complete in 35 min yielding 93 % of 1-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)ethanone **1a** (entry "Method B", Table 1). When this reaction was attempted at lower temperatures, 40 and 60 °C, these required longer reaction times for completion. Therefore, task-specific basic ionic liquid [bmim]OH was chosen as optimum system at 80 °C to extend this protocol. A number of other substituted aryl azides also underwent 1,3-cycloaddition with acetylacetone successfully under these conditions to afford corresponding triazoles in excellent yields (**1b**–**1h**, "Method B").

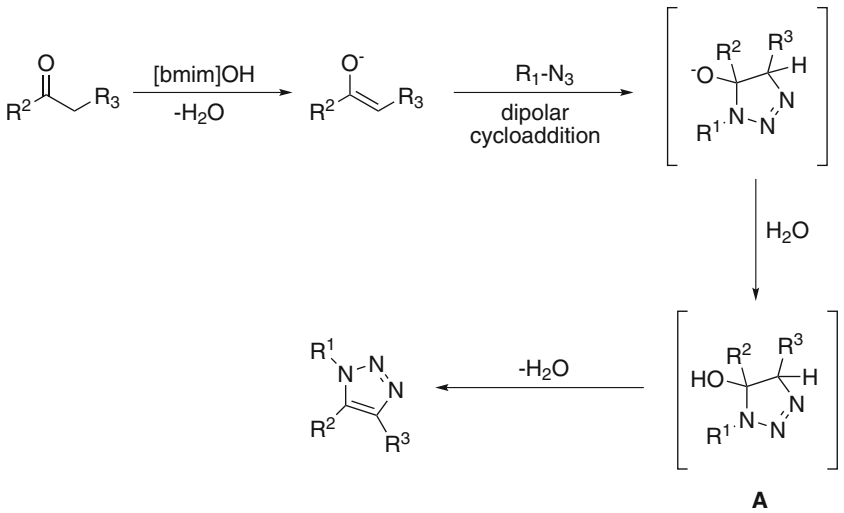
Similarly, the reactions of aryl azides (1.0 mmol) with ethyl acetoacetate (1.0 mmol) and methyl acetoacetate (1.0 mmol) under these conditions yielded corresponding ethyl 5-methyl-1-aryl-1*H*-1,2,3-triazole-4-carboxylates (**1i**–**1l**) and methyl 5-methyl-1-aryl-1*H*-1,2,3-triazole-4-carboxylate (**1m**–**1n**) in excellent yields. These results have been compiled in Table 1 (method B). The results indicate that reactions by both methods proceed fast and gave better yields with aryl azides having electron withdrawing groups compared to azides with electron donating groups. It can be inferred from the above results that method B is slightly superior as it requires shorter reaction times and the yields are slightly better. Since addition of aryl azides to active methylene compounds is catalyzed by base, therefore, ionic liquid [bmim]BF_4 acts as a reaction medium and L-proline acts as a base in method A, whereas in method B task-specific basic ionic liquid [bmim]OH functions both as the reaction medium and the base.

The plausible mechanism of the reaction catalyzed by basic ionic liquid [bmim]OH is outlined in Scheme 2. The reaction proceeds via initial formation of enolate in the presence of basic ionic liquid [bmim]OH, followed by Huiseng [3+2] cycloaddition of aryl azide to form triazoline intermediate **A**, which subsequently undergoes elimination to yield 1,4,5-trisubstituted-1,2,3-triazoles.

We have also investigated the recycling of basic ionic liquid [bmim]OH. We observed that [bmim]OH could be easily recovered after the completion of reaction and reused in subsequent runs using the model reaction of 4-nitrophenyl azide with acetylacetone in [bmim]OH at 80 °C. After completion of the reaction, it was cooled and water was added to the reaction mixture. The precipitate formed was collected by filtration at pump. The filtrate was concentrated under reduced pressure and dried to recover the ionic liquid for subsequent use. No significant loss in the yield of **1a** was observed after three cycles as **1a** was obtained in 93, 90 and 85 % yield after first, second and third cycle, respectively.

The cycloaddition of aryl azides to unsymmetrical ketones leads to different regioisomers. The reaction of various aryl azides by both methods ("Method A" and method B) is regioselective in nature and only one regioisomer was formed in case of unsymmetrical diketones as supported by the structure of cycloadduct **1k** which has been identified by X-ray diffraction analysis Fig. 1. The

Scheme 2 Plausible mechanistic pathway for [bmim]OH catalysed synthesis of 1,4,5-trisubstituted-1,2,3-triazoles



regioselectivity in the reaction arises because of selective approach of positively charged terminal nitrogen atom of aryl azide to electron-rich carbon atom of enolate during 1,3-dipolar addition, which resulted in a formation of only one favorable cyclic transition state with lower activation energy and hence only one regioisomer was formed.

Conclusion

In conclusion, we have reported efficient and environmentally benign methodologies for the synthesis of 1,4,5-trisubstituted-1,2,3-triazoles in ionic liquid [bmim]BF_4 in the presence of catalytic amount of L-proline and also in task-specific basic ionic liquid [bmim]OH. Corresponding triazoles were obtained in good-to-excellent yields by both methods. These methods offer advantages in terms of operational simplicity, easy work up, short reaction times and good yields as compared to previously reported methods.

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