

## One-pot synthesis of highly regioselective $\beta$ -azido alcohols catalyzed by Brønsted acidic ionic liquids

Sarvin Mohammadi-Aghdam, Hadi Jabbari, Omidali Pournalimardan, Faten Divsar, Issa Amini, Sami Sajjadifar\*

Department of Chemistry, Payame Noor University, P.O. BOX 19395-4697 Tehran, Iran

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### Abstract

In this protocol, 3-(2-carboxybenzoyl)-1-methyl-1H-imidazol-3-ium chloride [Cbmim]Cl and sulfonic acid functionalized pyridinium chloride [pyridine-SO<sub>3</sub>H]Cl as a new, reusable, and green Brønsted acidic ionic liquid (BAIL) catalyst were synthesized and successfully used for the one-pot ring-opening of epoxide with sodium azide (NaN<sub>3</sub>) in water at room temperature. Epoxides undergo ring-opening readily with NaN<sub>3</sub> in the presence of [Cbmim]Cl and [pyridine-SO<sub>3</sub>H]Cl to afford the corresponding  $\beta$ -azido alcohols as attractive and interesting materials in drug design and pharmaceuticals compounds in high yield with good regioselective under mild reaction conditions.

**Keywords:** [Cbmim]Cl; [pyridine-SO<sub>3</sub>H]Cl; epoxide; ring opening; Azidoalcohols; water.

### Introduction

Epoxides are the most useful synthetic middleman in organic synthesis [1]. Because of their ring strain and high reactivity, their reactions with various nucleophiles lead to high regio- and stereoselective ring-opening products [2-6]. Epoxides are widely used as building blocks in organic chemistry. Ring opening reactions of epoxide with nucleophiles are considered as an interesting approach in organic synthesis of many functionalized oxygenated compounds. The reaction with nucleophiles such as oxygen compounds. (Water, alcohols and phenols) [7].

The reaction of azide ion with epoxides, in the presence of a catalyst, is a suitable method for the preparation

of  $\beta$ -Azidoalcohols. Azidoalcohols are important categories of organic compounds as they serve in the synthesis of aminoalcohols, amino sugars and carbocyclic nucleosides [8-11].  $\beta$ -Azidoalcohols would also be regarded as useful compounds in organic synthesis as either precursors of 2-amino alcohols or in the chemistry of carbohydrates, nucleosides, lactams, and oxazolines [12]. These compounds are versatile intermediates in organic synthesis and increasingly important in drugs and pharmaceuticals [13,14]. The ring-opening of epoxides, which has been regarded as innovative ways to obtain the direct azidolysis of epoxides in the presence of sodium azide, is frequently performed under several different conditions [15-19].

\*Corresponding author: Sami Sajjadifar

Tel: +98 (84) 32228316, Fax: +98 (84) 32221053

E-mail: sami.sajjadifar@gmail.com

But some of these methods are limited to specific epoxides and are not applicable as versatile reagents in the preparation of azidohydrines. In this sense, it is worth mentioning that these methods suffer from some disadvantages such as long reaction times, low regioselectivity, using expensive catalysts and difficulty in preparation of catalysts, difficulty in work-up and isolation of products. Therefore, it seems that there is still a need for development of green methods that proceed under green and eco-friendly conditions.

During the course of the last two decades, the application of Ionic liquids (ILs) as attractive and interesting materials has been more and more increased, because of their unique properties including their non-flammability, high thermal and chemical stability, wide liquid-state temperature range, large electrochemical window, non-volatility, and favorable solvation behaviors. ILs have been emerged widely for various areas; such as ease of separation, electrochemistry processes, spectroscopy, and extraction. Moreover, they are used as catalysts, reagents, and solvents in a variety of organic transformation [20-24]. Among different kinds of ionic liquids, Brønsted acidic ionic liquids (BAILs) have the greatest potential for the development of environmentally friendly acid catalysts in synthetic organic chemistry. It is due to the fact that they effectively combine the advantages of liquid and solid acids and provide high levels of operational simplicity, efficacy, and selectivity coupled with a low environmental impact [25-27]. Besides, application of acidic ionic liquids (AILs) was reported in some literatures [28-33].

Organic azides have been important compounds at the interface between chemistry, biology, medicine, and materials science.  $\beta$ -Azidoalcohols as attractive and interesting materials have been more and more increased, because of their compounds, the main target in medicinal chemistry is to synthesize compounds for drug discovery or pharmaceuticals compounds [34,35]. Azide groups are high potential targets for drug discovery and biologically active in natural compounds such as anti-allergic, anti-microbial, and anti-HIV properties [36]. Triazoles derivatives which are regarded as subclass of azido compound possess a number of desirable features in the context of medicinal chemistry.  $2\beta$ -,  $6\beta$ - and  $16\beta$ -azido-androstanes synthesis from ring-opening of epoxide with sodium azide via the 'click reaction' of ferrocenyl azides and steroids with terminal alkynes [37]. Glycerol 3-phosphate acyltransferase (GPAT) inhibitors synthesized by multi step, regio- and stereoselectively ring-opening of epoxide with sodium azide to produce azidoalcohols, reduced and coupled to octane sulfonyl chloride to yield sulfonamides under reflux condition [38].

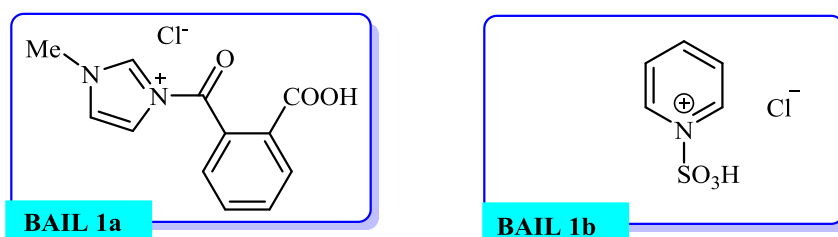
Recently ionic liquids have been successfully employed as dual reagents (solvents + catalysts) for a variety of reactions, but their use as catalyst under solvent-free conditions needs to be given more attention [39-40]. Ionic liquids have been widely vaunted as greener reagents, suitable for a range of organic reactions and providing possibilities such as, enhanced rate and reactivity, control of product distribution, ease of product recovery and recycling.

In this protocol, 3-(2-carboxybenzoyl)-1-methyl-1H-

imidazol-3-ium chloride [Cbmim]Cl and sulfonic acid functionalized pyridinium chloride [Pyridine-SO<sub>3</sub>H]Cl as a new heterogeneous acidic and Brønsted acidic ionic liquid catalyst were designed and synthesized from inexpensive and commercially available starting materials (Scheme 1).

In continuation of our search for new catalysts [41-44], we describe an

efficient protocol for the selective azidolysis of epoxides using NaN<sub>3</sub> in the presence of [Cbmim]Cl and [Pyridine-SO<sub>3</sub>H]Cl as a new heterogeneous acidic catalyst. This method has advantages, including the use of an inexpensive, reusable and eco-friendly ionic liquid, short reaction times, high yields, and simplicity of the product isolation.



**Scheme 1.** Synthesis of BAIL **1a** [Cbmim]Cl and BAIL **1b** [Pyridine-SO<sub>3</sub>H]Cl

## Experimental

### General

All chemicals were purchased from Merck or Fluka Chemical Companies. The products were characterized by comparing their spectroscopic data (<sup>1</sup>H NMR, <sup>13</sup>C NMR and IR) and physical properties with those reported in the literature. NMR spectra were recorded in CDCl<sub>3</sub> on a Bruker advanced DPX 500 and 400 MHz instrument spectrometers using TMS as internal standard. IR spectra were recorded on a Frontier FT-IR (Perkin Elmer) spectrometer using a KBr disk. All yields refer to isolated products. Progress of the reactions and the purity check of the final products were carried out by thin layer chromatography (TLC) using silica gel precoated aluminium sheets (60–120 mesh; Merck) and visualization with ultraviolet light at 365 and 254 nm.

### General procedure for the synthesis of [Cbmim]Cl BAIL **1a** and [Pyridine-SO<sub>3</sub>H]Cl BAIL **1b**

3-(2-Carboxybenzoyl)-1-methyl-1H-imidazol-3-ium chloride [Cbmim]Cl

BAIL **1a** and sulfonic acid functionalized pyridinium chloride [Pyridine-SO<sub>3</sub>H]Cl BAIL **1b** were prepared according to the literature procedure [47-48].

### Reactions of epoxides with NaN<sub>3</sub> catalyzed by [Cbmim]Cl BAIL **1a**

[Cbmim]Cl BAIL **1a** (5 mol%) was added to a mixture of the epoxide (1 mmol) and NaN<sub>3</sub> (3 mmol) in water (5 mL). The reaction mixture was magnetically stirred at room temperature for the appropriate time. Progress of the reaction was monitored by TLC using ethyl acetate: *n*-hexane (1:5). After completion of reaction, the mixture was extracted with diethyl ether (3×5 mL), washed with brine, dried with CaCl<sub>2</sub> and evaporated under reduced pressure. The desired azidoalcohols were obtained in good to excellent isolated yields (85-95%).

### Reactions of epoxides with NaN<sub>3</sub> catalyzed by [Pyridine-SO<sub>3</sub>H]Cl BAIL **1b**

[Pyridine-SO<sub>3</sub>H]Cl BAIL **1b** (3 mol%) was added to a mixture of the epoxide (1 mmol) and NaN<sub>3</sub> (3 mmol) in water

(5 mL). The reaction mixture was magnetically stirred at room temperature for the appropriate time. Progress of the reaction was monitored by TLC using ethyl acetate: *n*-hexane (1:5). After completion of reaction, the mixture was extracted with diethyl ether (3×5 mL), washed with brine, dried with CaCl<sub>2</sub> and evaporated under reduced pressure. The desired azidohydrines was obtained in good to excellent isolated yields (86-94%).

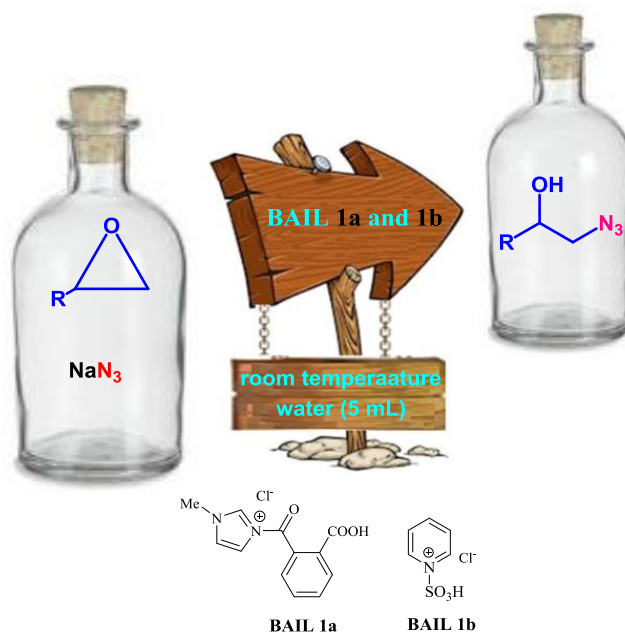
### Results and discussion

In recent years, ionic liquids have emerged as other reaction media for the immobilization of transition metal based catalysts, Lewis acids and enzymes. They are solvents with unique properties such as tunable polarity, high thermal stability, immiscibility with a number of organic solvents, insignificant vapour pressure and ease of recyclability. They are referred to as designer solvents as their properties such as hydrophilicity, hydrophobicity, Lewis acidity, viscosity and density can be changed by the fine tuning of parameters such as the choice of organic cation, inorganic anion and the

length of alkyl chain attached to the organic cation.

The structure of BAIL **1a** [Cbmim]Cl was identified by studying its FT-IR, <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra (supplementary data) [47]. Then, the structure of BAIL **1b** [Pyridine-SO<sub>3</sub>H]Cl was identified by studying its FT-IR, <sup>1</sup>H NMR, <sup>13</sup>C NMR, UV, TGA, DTGA, and MS spectra [48]. BAIL **1a** [Cbmim]Cl was easily prepared from the reaction between phthalic anhydride and 1-methyl imidazole in the presence of iodine for 20 min, and the final acidification step was performed by HCl (37%). Also, BAIL **1b** [Pyridine-SO<sub>3</sub>H]Cl was easily prepared from the reaction between pyridine and chlorosulfonic acid in dry-dichlorometane at 0 °C.

In order to evaluate the catalytic potential of the prepared BAIL **1a** [Cbmim]Cl and BAIL **1b** [Pyridine-SO<sub>3</sub>H]Cl as a new heterogeneous acidic catalyst in organic transformations, we decided to examine its activity in one-pot preparation of β-azido alcohols derivatives (Scheme 2).



**Scheme 2.** Reactions of epoxides with NaN<sub>3</sub> catalyzed by acidic ionic liquid

Firstly, we carried out the reaction between phenyl glycidyl ether (1 mmol) with sodium azide (1.5 mmol) in the presence of various amounts of BAIL **1a** [Cbmim]Cl as model reaction for the optimization of the reaction conditions (Table 1). As evident from Table 1, in the absence of BAIL **1a** [Cbmim]Cl, the product is not obtained

(Table 1, Entry 1). According to the reaction yield (91%) and reaction time (4 min), quantity 5% of BAIL **1a** [Cbmim]Cl is suitable (Table 1, Entry 4). Also, no improvement is observed in the yield and reaction time when increasing the amount of BAIL **1a** [Cbmim]Cl (Table 1, Entry 5).

**Table 1.** Result of amount of BAIL **1a** [Cbmim]Cl in the synthesis of 2-azido-2-phenyl-1-ethanol under solvent-free conditions and room temperature

Entry	Catalyst loading (%)	Reaction time (min)	Yield (%) <sup>a</sup>
1	Catalyst-free	24 h	Trace
2	1	20	75
3	3	10	89
4	5	4	91
5	10	10	84

<sup>a</sup>Isolated yield

In the next study, we carried out the reaction between phenyl glycidyl ether (1 mmol) with sodium azide (1.5 mmol) using 5 mol% of BAIL **1a** [Cbmim]Cl in some solvents (5 mL), i.e. tetrahydrofuran, chloroform, dichloromethane, ethyl acetate, acetonitrile and water as model reaction for optimization of the reaction conditions (Table 2). The results are summarized in Table 2. As evident from Table 2, according to the yield

and reaction time, among the listed solvents, water is the best solvent for the reaction (Table 2, Entry 6). Water is a desirable solvent for chemical reactions for reasons of cost, safety and environmental concerns. In this regard, the use of water in this reaction gave only greater regioselectivity to the ring-opening of epoxide.

**Table 2.** Result of various solvents in the synthesis of 2-azido-2-phenyl-1-ethanol at room temperature

Entry	Solvent	Reaction time (min)	Yield (%) <sup>a</sup>
1	THF	10	46
2	CHCl <sub>3</sub>	15	58
3	CH <sub>2</sub> Cl <sub>2</sub>	19	53
4	CH <sub>3</sub> CO <sub>2</sub> Et	20	61
5	CH <sub>3</sub> CN	10	66
6	H <sub>2</sub> O	4	95

<sup>a</sup>Isolated yield

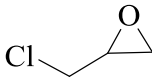
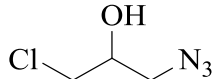
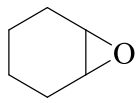
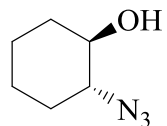
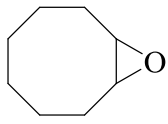
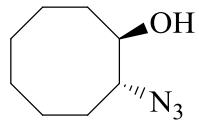
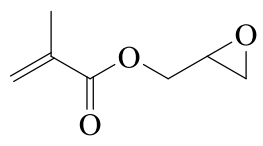
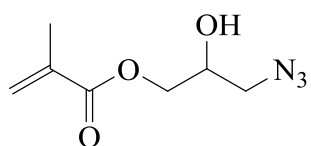
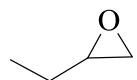
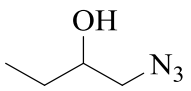
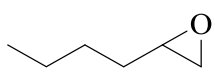
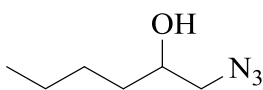
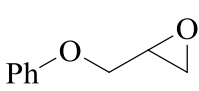
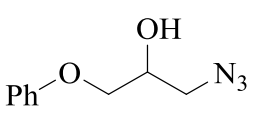
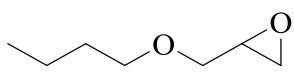
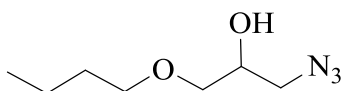
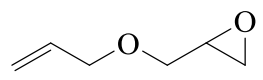
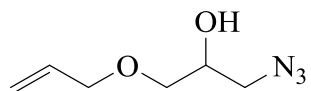
Subsequently, under optimal conditions, 1 mmol epoxide: 3 mmol azide: 5 mol% BAIL **1a** or 3 mol% BAIL **1b** and 5 mL water at room temperature, the generality and synthetic scope of this coupling protocol were demonstrated by synthesizing a series of 2-azidoalcohols in excellent yield and short reaction time

In case of epichlorohydrin (Table 3, Entry 2), the diazido alcohol is formed as a product. In cases of cyclic epoxides

(Table 3, Entries 3 and 4), *trans*-products are formed. Obviously, in these reactions, the attack appears to be largely, if not entirely, at the primary carbon atom of the epoxide ring. The orientation of ring-opening is characteristically observed for reactions of monoalkyl-substituted epoxides under S<sub>N</sub><sup>2</sup> conditions and is probably dictated by steric and electronic factors.

**Table 3.** Preparation of β-azido alcohols from various epoxides with sodium azide anion in the presence BAIL **1a** [Cbmim]Cl in water

Entry	Epoxide	product	Time (min)	Yield (%) <sup>a</sup>
1		+	2	88 (11:89)

2			6	85
3			7	90
4			7	87
5			6	90
6			5	93
7			8	89
8			4	95
9			6	90
10			5	89

<sup>a</sup>Isolated yield

In another study, after the success of BAIL **1a** [Cbmim]Cl for the synthesis of  $\beta$ -azido alcohols, we decided to explore the catalytic activity of BAIL **1b** [Pyridine-SO<sub>3</sub>H]Cl as a new heterogeneous acidic catalyst for the synthesis of  $\beta$ -azido alcohols. For this purpose, to optimize the reaction

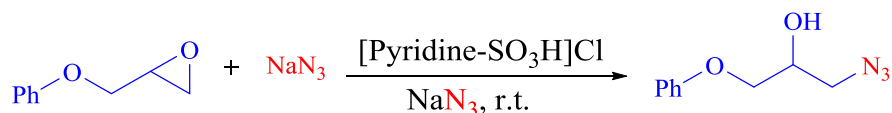
conditions, we carried out the reaction between phenyl glycidyl ether (1 mmol) with sodium azide (1.5 mmol) in the presence various amounts of BAIL **1b** [Pyridine-SO<sub>3</sub>H]Cl as the model reaction for the optimization of the reaction conditions (Table 4). As evident from Table 4, In the absence of



**BAIL1b** [Pyridine-SO<sub>3</sub>H]Cl, the product is not obtained (Table 4, Entry 1). According to the reaction yield (92%) and reaction time (9 min), quantity 3 mol% of **BAIL 1b** [Pyridine-SO<sub>3</sub>H]Cl, is suitable (Table 4,

Entry 4). Also, no improvement is observed in the yield and reaction time when increasing the amount of **BAIL 1b** [Pyridine-SO<sub>3</sub>H]Cl (Table 4, Entries 4 and 5).

**Table 4.** Result of amount of **BAIL1b** [Pyridine-SO<sub>3</sub>H]Cl in the synthesis of 2-azido-2-phenyl-1-ethanol under solvent-free conditions at room temperature



Entry	Catalyst loading (mol%)	Reaction time (min)	Yield (%) <sup>a</sup>
1	Catalyst-free	24 h	Trace
2	1	45	72
3	2	35	81
4	3	18	92
5	4	15	84
6	5	15	85

<sup>a</sup>Isolated yield

In the next study, we carried out the reaction between phenyl glycidyl ether (1 mmol) and sodium azide (1.5 mmol) using 3 mol% of **BAIL1b** [Pyridine-SO<sub>3</sub>H]Cl in some solvents (5 mL), i.e. tetrahydrofuran, chloroform, dichloromethane, ethyl acetate, acetonitrile and water as the model

reaction for optimization of the reaction conditions (Table 5). The results are summarized in Table 5. As evident from Table 5, according to the yield and reaction time, among the solvents listed, water is the best solvent for the reaction (Table 5, Entry 6).



**Table 5.** Result of various solvents in the synthesis of 2-azido-2-phenyl-1-ethanol at room temperature

Entry	Solvent	Reaction time (min)	Yield (%) <sup>a</sup>
1	THF	25	51
2	CHCl <sub>3</sub>	20	48
3	CH <sub>2</sub> Cl <sub>2</sub>	20	41
4	CH <sub>3</sub> CO <sub>2</sub> Et	35	45
5	CH <sub>3</sub> CN	45	75
6	H <sub>2</sub> O	10	94

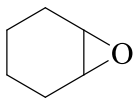
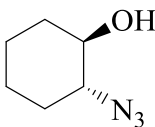
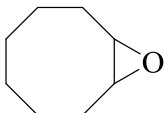
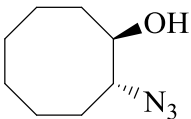
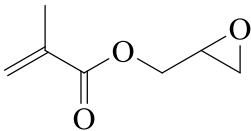
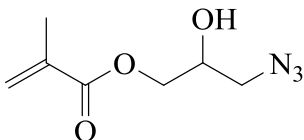
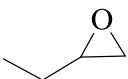
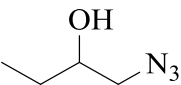
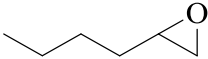
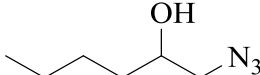
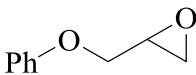
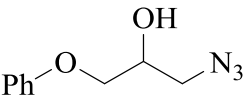
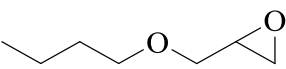
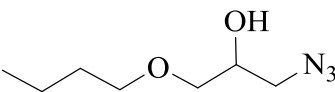
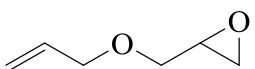
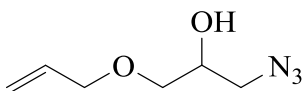
<sup>a</sup>Isolated yield

After identifying the optimum reaction conditions, the condensation reaction between phenyl glycidyl ether (1 mmol) with sodium azide (1.5 mmol) in the presence of **BAIL1b**

[Pyridine-SO<sub>3</sub>H]Cl (3 mol%) in water at room temperature was chosen as a model reaction. All the 2-azidoalcohols were obtained in excellent yields and short reaction time (Table 6).

**Table 6.** Preparation of  $\beta$ -azido alcohols from various epoxides with sodium azide anion in the presence **BAIL 1b** [Pyridine-SO<sub>3</sub>H]Cl in water

Entry	Epoxide	Product	Time (min)	Yield (%) <sup>a</sup>
1			5	90 (11:89)
2			15	87

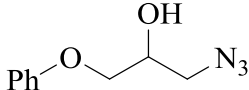
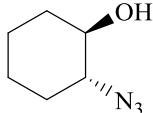
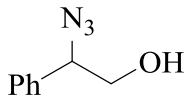
3			18	88
4			20	90
5			15	90
6			12	91
7			18	86
8			10	94
9			15	92
10			12	87

<sup>a</sup>Isolated yield

As it is evident from Table 7, BAIL **1a** [Cbmim]Cl and BAIL **1b** [Pyridine-SO<sub>3</sub>H]Cl as a new heterogeneous acidic catalyst afforded good results for the regioselective ring-opening of some epoxides such as 2-(phenoxymethyl)oxirane, 2-phenyloxirane, and cyclohexene epoxide in comparison to the other catalysts. In order to evaluate the

efficiency of our introduced method, more recently developed methods were compared to our present method on the basis of the yields and reaction times parameters, the results are given in Table 7. As is evident, the BAIL **1a** [Cbmim]C and BAIL **1b** [Pyridine-SO<sub>3</sub>H]Cl improve the synthesis of product.

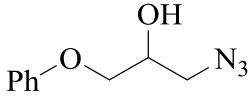
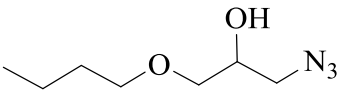
**Table 7.** Comparison of results for synthesis of model product by BAIL **1a** [Cbmim]Cl and BAIL **1b** [Pyridine-SO<sub>3</sub>H]Cl with those obtained using other reported catalysts

Entry	Conditions	Time (min)	Yield (%)	Product
1	(TBA) <sub>4</sub> PFeW <sub>11</sub> O <sub>39</sub> .3H <sub>2</sub> O, CH <sub>3</sub> CN:H <sub>2</sub> O, 80 °C	3 h	94	
	Network polymer, water, 80 °C	90	89	
	[Hmim]N <sub>3</sub> , CH <sub>3</sub> CN, 60 °C	55	94	
	Polyethylene glycol 300, 60 °C	1 h	90	
	Fe <sub>3</sub> O <sub>4</sub> @SiO <sub>2</sub> /BNC, water, 90 °C	50	95	
	[Cbmim]Cl, water, room temperature	4	95	
	[Pyridine-SO <sub>3</sub> H]Cl, water, room temperature	10	94	
2	(TBA) <sub>4</sub> PFeW <sub>11</sub> O <sub>39</sub> .3H <sub>2</sub> O, CH <sub>3</sub> CN:H <sub>2</sub> O, 80 °C	2 h	93	
	Network polymer, water, 80 °C	80	83	
	[Hmim]N <sub>3</sub> , CH <sub>3</sub> CN, 60 °C	65	83	
	Polyethylene glycol 300, 60 °C	1 h	85	
	Fe <sub>3</sub> O <sub>4</sub> @SiO <sub>2</sub> /BNC, water, 90 °C	20	88	
	[Cbmim]Cl, water, room temperature	7	90	
	[Pyridine-SO <sub>3</sub> H]Cl, water, room temperature	18	88	
3	(TBA) <sub>4</sub> PFeW <sub>11</sub> O <sub>39</sub> .3H <sub>2</sub> O, CH <sub>3</sub> CN:H <sub>2</sub> O, 80 °C	4.5 h	85	
	Network polymer, water, 80 °C	1.5	85	
	[Hmim]N <sub>3</sub> , CH <sub>3</sub> CN, 60 °C	50	88	
	Polyethylene glycol 300, 60 °C	1.5	85	
	Fe <sub>3</sub> O <sub>4</sub> @SiO <sub>2</sub> /BNC, water, 90 °C	15	95	
	[Cbmim]Cl, water, room temperature	2	88	
	[Pyridine-SO <sub>3</sub> H]Cl, water, room temperature	5	90	

Based on another study, we checked the recovery and reusability of the BAIL **1a** [Cbmim]Cl and BAIL **1b** [Pyridine-SO<sub>3</sub>H]Cl. For this purpose, the reaction between 2-(phenoxyethyl)oxirane and sodium azide (Table 3, Entry 8) in the presence of BAIL **1a** [Cbmim]Cl and also the reaction between 2-

(butoxymethyl)oxirane and sodium azide (Table 6, Entry 9) in the presence of BAIL **1b** [Pyridine-SO<sub>3</sub>H]Cl were selected as a model reaction. As evident from Table 8, the activity of the BAIL **1a** [Cbmim]Cl and BAIL **1b** [Pyridine-SO<sub>3</sub>H]Cl were slightly decreased after reuse six times.

**Table 8.** Reusability of the BAIL **1a** [Cbmim]Cl and BAIL **1b** [Pyridine-SO<sub>3</sub>H]Cl in the reaction of 2-(phenoxyethyl)oxirane and 2-(butoxyethyl)oxirane with sodium azide

Product	Yield (%) Cycle 1	Yield (%) Cycle 2	Yield (%) Cycle 3	Yield (%) Cycle 4	Yield (%) Cycle 5	Yield (%) Cycle 6
	95	94	92	90	90	88
	92	91	91	89	87	87

### Conclusion

In summary, we have introduced 3-(2-carboxybenzoyl)-1-methyl-1H-imidazol-3-ium chloride [Cbmim]Cl and sulfonic acid functionalized pyridinium chloride [pyridine-SO<sub>3</sub>H]Cl as a new, reusable, and green Brønsted acidic ionic liquid (BAIL) catalyst for the preparation of  $\beta$ -azido alcohols.  $\beta$ -azido alcohols were obtained using 3-5 mol% of BAIL **1a** and **1b** at room temperature in water as a green solvent. Other promising points for this method are short reaction time, high yield of products, recovery and reusable of BAIL **1a** and **1b**, non-toxic, and low cost.

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