

Synthetic Strategies of Ionic Liquids an Emerging Green Material for Many Applications in Material Science

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www.jrasb.com || June 2026

Date of Submission: 01-06-2026

Date of Publication: 12-06-2026

ABSTRACT

Ionic Liquids are new emerging monomers or polymer molecules work as green materials and are best known for various applications in material sciences. Suitable ILs are designed with explicit features to perform specific functions. That can be used in electrochemical applications such as electrolytes, solvents in Lithium-ion batteries, solvents in fuel cells superconductors and solar cells. In this article we are focussing on synthesis of some interesting and widely used ionic liquids. The synthetic applicability of these smart green materials is paved ways in organic synthesis. They can behave as an excellent solvent as well as catalyst for such transformations. Here we are highlighting the synthesis of green designer materials which is based on selection of specific cation and anions for ILs that perform specific function and possess certain specific desirable properties.

Keywords- Ionic liquid; Green designer material; Catalyst; Environment benign.

I. INTRODUCTION

The most crucial tool available nowadays to reduce environmental risks is green chemistry, a developing area of science. It addresses a much wider variety of challenges and can help create environmentally friendly reaction conditions, minimise energy use, and design compounds that are naturally safer and more affordable.

Ionic liquids (ILs) are defined as salts composed of organic cations and anions. These compounds have the unique property of melting at temperatures below 100° C. ILs are known to contain small amounts of water approximately 200–600 ppm, which can act as nucleophile in reactions. Because IL have the freedom to select the organic cations and anions, they are adjustable and are known as designer solvents.

ILs have diverse applications due to their favorable characteristics, including ease of handling, processing, thermal and chemical stability, short reaction time, high yields, efficiency, recyclability, and reusability.^{i,ii} They find use in the production of advanced materials,ⁱⁱⁱ formulation of vaccines, small molecule drugs,^{iv} topical^{v,vi,vii} and transdermal drug delivery systems.^{viii}

Ionic Liquids are presented as a clean, efficient, and eco-friendly substitute for conventional organic solvents. Their unique thermal, physical, chemical and biological properties make them attractive for a wide range of applications in chemical research. ILs can serve as effective catalysts, supporting the development, adjustment and reactivity of reagents and intermediates in multicomponent transformations.^{ix} Functionalized ionic liquids can be synthesized to offer advantages over existing catalysts. Furthermore, ILs can be recovered after use as catalysts with minimal loss in catalytic activity.^x Ionic liquids (ILs) are gaining recognition as eco-sustainable solvents, particularly as replacements for traditional mediums in chemical reactions. One of the significant advantages of ILs is their incredibly low vapor pressure. This process is easy to maintain and keep within a process. While there are multiple advantages to utilizing ILs, there are still limitations that restrict their full applicability. To overcome the limitations of ILs, a strategy was introduced i.e. immobilizing or supporting ILs on solid support materials. This approach combines the favorable characteristics of ILs such as their low volatility and high solvent capacity, with the benefits of using solid support materials in reactions.^{xi}

II. DESIGNER SOLVENTS - FLEXIBILITY OF CHOOSING FUNCTIONAL GROUPS TO MAKE IONIC LIQUIDS

Ionic liquids are considered as designer solvents because they can be selectively designed by choosing required components that constitute its molecule i.e., by selective cations and anions. Thus, the flexibility of its chemical and physical properties can be maintained based on the requirement of experiments designed.^{xii} Some of the important component cations and anions are discussed below.

2.1. Common Cations and Common Anions:

Formulation of ionic liquids rely on common ionic liquids including those containing imidazolium, pyridinium, pyrrolidinium, ammonium, phosphonium cations and anions such as Cl^- , Br^- , BF_4^- . Cations containing both saturated and unsaturated heterocycles are common.

2.1. a. Organic cations

N,N'-dialkyl imidazolium (I), *N*-alkylpyridinium(II), *N,N*-dialkylpyrrolidinium(III), Tetra alkyl ammonium (IV), Alkylphosphonium(V), Alkylsulfonium(VI), Alkylnitride (VII), Dialkylpyrazolium(VIII), Alkyl thiazolium (IX) (Figure: 1).

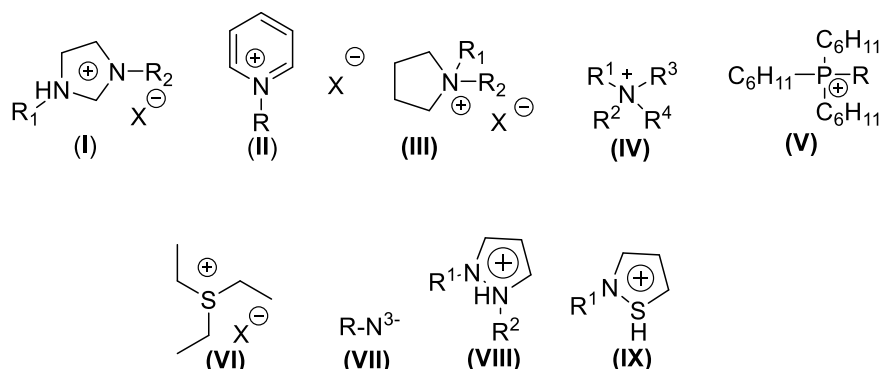


Figure: 1

2.1.b. Anions

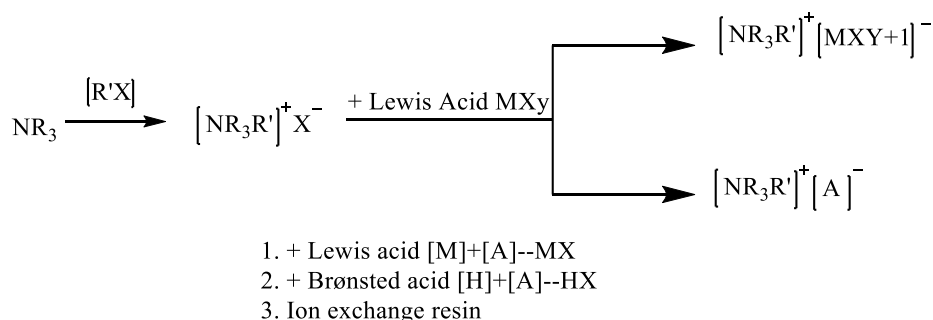
Halide-based (Cl^- , Br^- , F^-), Halogeno-based (BF_4^- , PF_6^- , PF_6^-), Halogenoaluminium(III), Carborane anions are the most common and selected anions for designing ionic liquids.

III. IONIC LIQUID AS CATALYST WITHOUT AND WITH SOLID SUPPORT

Ionic liquids can perform dual functions as catalyst and at the same time as a solvent system in many organic transformations.^{xiii, xiv} Though it can work as an efficient catalyst with or without solid support, many solid supports are available for fabrication and use as recyclable, reusable catalyst for organic transformations^{xv}.

IV. APPROACH APPLIED FOR SYNTHESIS OF IONIC LIQUID

The general synthetic procedure for the synthesis of ionic liquids can be achieved through the formation of the desired cation and the anion exchange. The desired cation can be synthesized either by the protonation of the amine by an acid or through quaternization reactions of amine with a haloalkane and heating the mixture. Anion exchange reactions can be carried out by treatment of halide salts with Lewis acids to form Lewis acid-based ionic liquids or by anion metathesis.^{xvi} (Scheme 1)

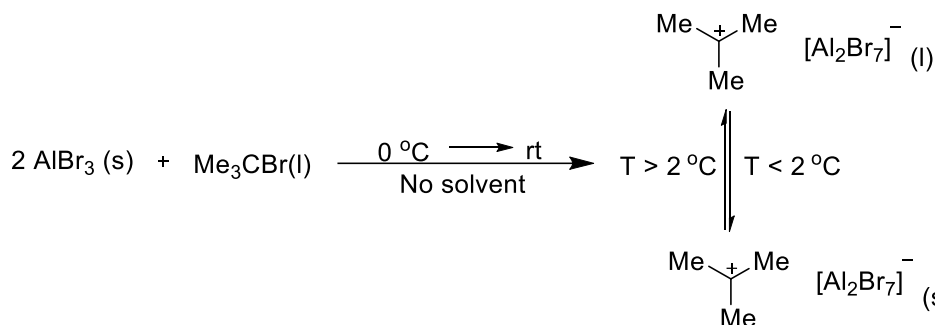


(Scheme 1) :Synthesis paths for the ILs

V. VARIOUS IONIC LIQUIDS AVAILABLE

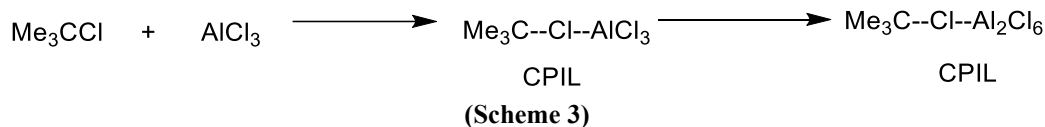
5.1. [(Me)₃C]⁺[Al₂Br₇]⁻ Ionic Liquid

[(Me)₃C]⁺[Al₂Br₇]⁻ was formed from tert-butyl bromide using aluminium bromide and in the absence of a solvent (Scheme 2). The low nucleophilicity of [Al₂Br₇]⁻ ion stabilises the tert-butyl cation and therefore considered as a convenient source of Bronsted acidic tert-butyl cation.^{xvii}



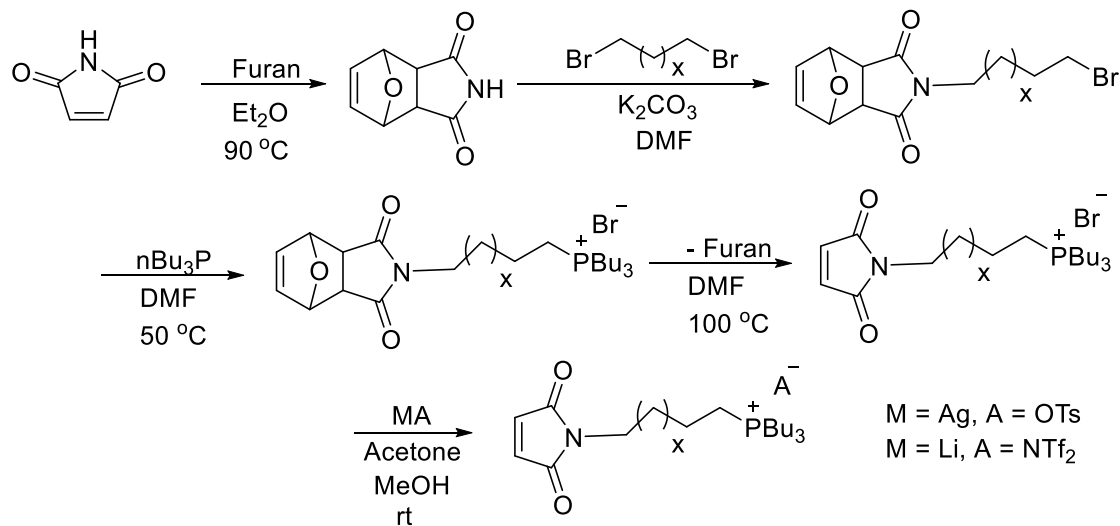
(Scheme 2)
5.2. Carbonium Pseudo Ionic Liquid
Carbonium pseudo ILs (CPILs) resemble ionic liquids with metal complex but have a

different composition (Scheme 3). Butyl halides make the hard acid AlCl₃ soft to form highly polarized liquids.^{xviii}



5.3. Maleimide-modified phosphonium ionic liquids

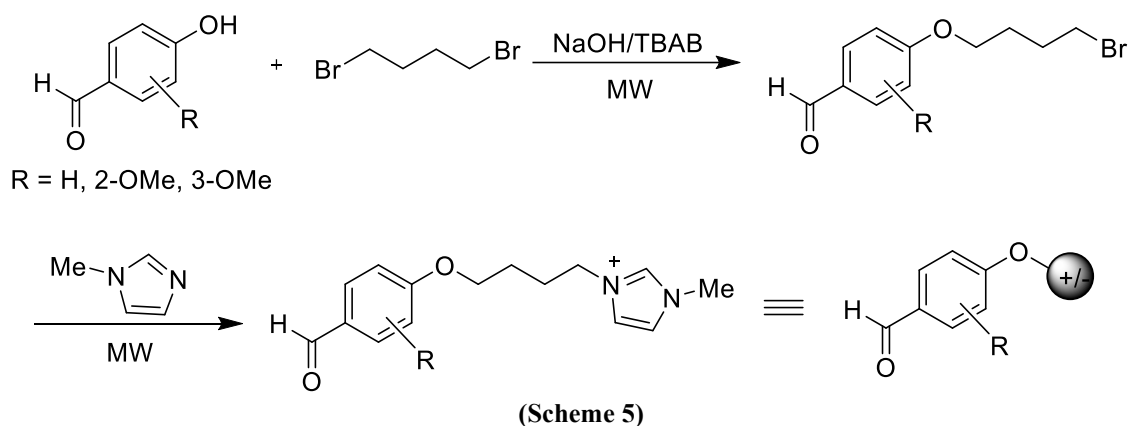
To create TSIL the structure of the IL was altered to include *N*-substituted maleimide. Shown below is the synthesis of maleimide-modified phosphonium ionic liquids (Scheme 4).^{xix}



(Scheme 4)

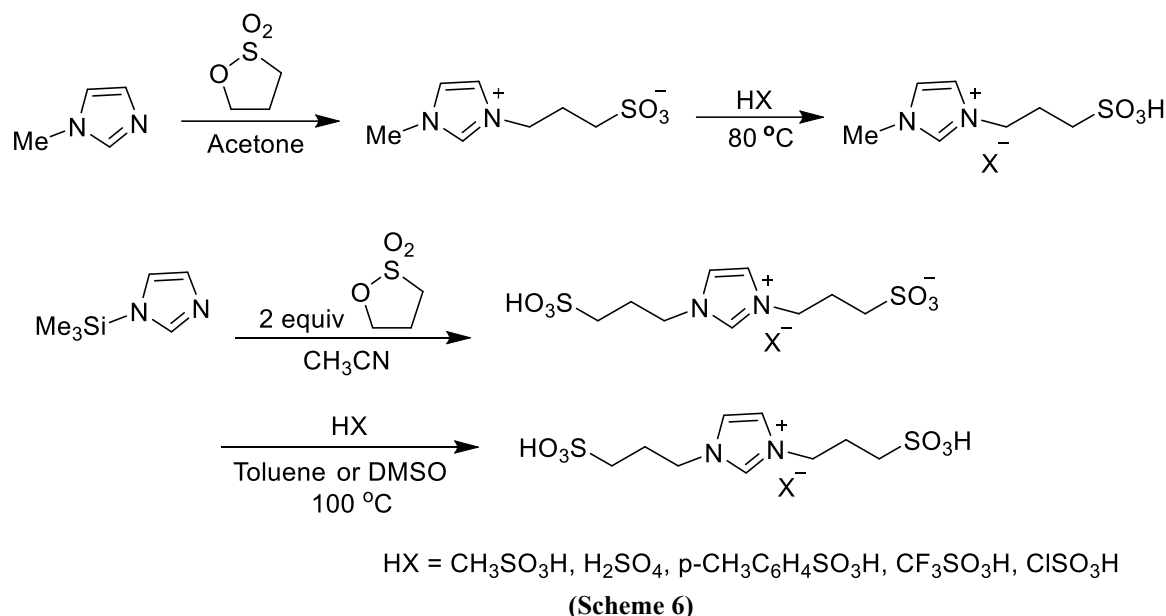
5.4. Benzaldehyde-functionalized ionic liquids

N-alkylimidazole was quaternized with benzaldehyde-functionalized alkyl bromides to form the benzaldehyde-functionalized ionic liquids on being exposed to microwave radiation^{xx} (Scheme 5).



5.5. Synthesis of Ionic Liquids based on Sulphonic acids:

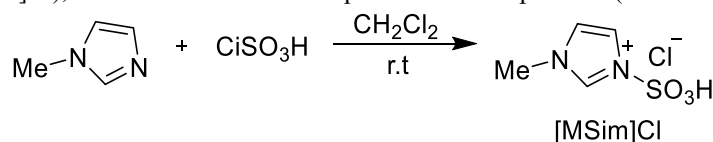
Strong acids (HX) were used to protonate the sulfonate anion and create the ionic liquids that contain mono- and bis-sulfonic acids, respectively. These acids included chlorosulfonic acid, methanesulfonic acid, sulfuric acid, trifluoromethanesulfonic acid and p-toluenesulfonic acid. (Scheme 6).^{xxi}



5.6. Imidazolium based IL:

5.6.1. 3-Methyl-1-sulfonic acid imidazolium chloride ([Msim]Cl) Ionic Liquid

1-methylimidazole and chlorosulfonic acid on combination resulted in the formation of 3-methyl-1-sulfonic acid imidazolium chloride ([Msim]Cl), a bronsted acidic ionic liquid at room temperature (Scheme 7).^{xxii}

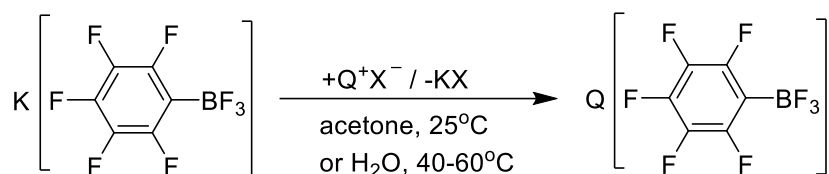


5.6.2. Imidazolium based trifluoroborates for ionic liquids with specific properties

A unique synthetic platform for ionic liquids (ILs) has been presented that is based on the polyfluorophenyltrifluoroborate anions, $Q[4-XC_6F_4BF_3]$ ($X = F, RO$). By ion exchange (metathesis) reactions of $Q^+ X^-$ ($X = Cl, Br$; $Q = Et_4N$ or directly by nucleophilic substitution of BMIM $[C_6F_6BF_3]$), new ionic liquids can be made at high yields (Scheme 8). The starting chemicals for the reactions were easily soluble in solvents like acetonitrile or acetone, but the potassium halide was not. Alkali metal chlorides ions were desirable starting materials because they dissolve in acetone less

readily (by 200–300 times less than similar bromides) than do bromides, resulting in halogen-free ionic liquids that may be removed by dissolving the reaction mixture in dichloromethane.

The physicochemical properties of ILs were influenced by the sort of substituents present in the fluorinated ring. The fundamental properties of ionic liquids controlled by the Coulomb interaction and the formation of hydrogen bonds between the fluorine atoms of the BF_3 moiety and the hydrogen atom H-2 of the imidazolium cation. This opens up a variety of possibilities for their use as functional materials in many fields.^{xxiii}

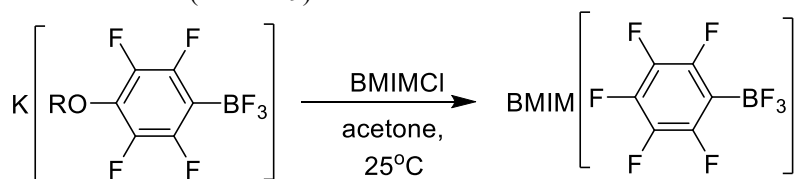


Q = Et_4N (a), EMIM (b), BMIM (c), OMIM (d), BMMIM (e); X = Cl, Br

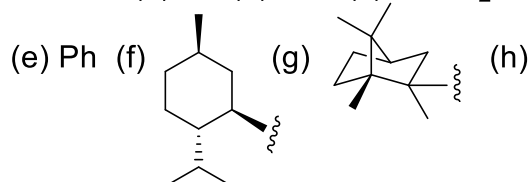
Preparation of the ionic liquids based on the $\text{C}_6\text{F}_5\text{BF}_3$ anion.

(Scheme 8)

BMIMCl reacted with the necessary salts resulting in the formation of 2,3,5,6-tetrafluorophenyltrifluoroborates 1-butyl-3-methylimidazolium using acetone. The replacement of the p-fluorine atom in the pentafluorotrifluoroborate anion with the proper nucleophiles^{xxiv} resulted in the production of the salts. *L*(-)-isopulegol and *L*(-)-borneol formed the chiral salts g and h following the same method. (Scheme 9)



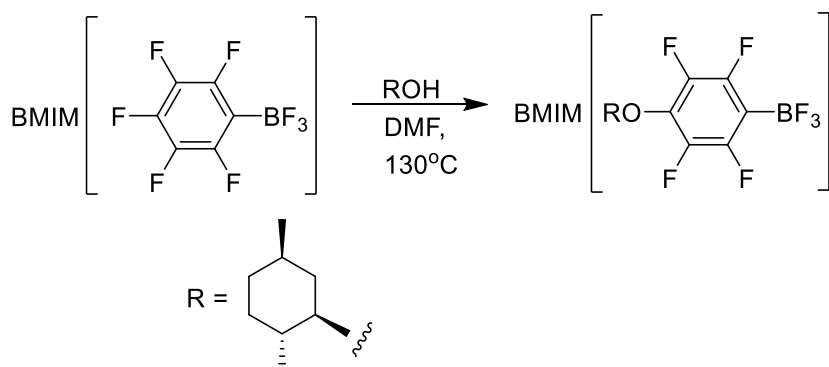
R = Me (a) Pr (b) t-Bu (c) PhCH_2CH_2 (d) PHCH_2



Preparation of the ionic liquids based on the $[4\text{-ROC}_6\text{F}_4\text{-BF}_3]$ -anions.

(Scheme 9)

BMIM $[\text{C}_6\text{F}_5\text{BF}_3]$ undergoes direct conversion into ionic liquids either through nucleophilic substitution or use of appropriate reagent (Scheme 10).

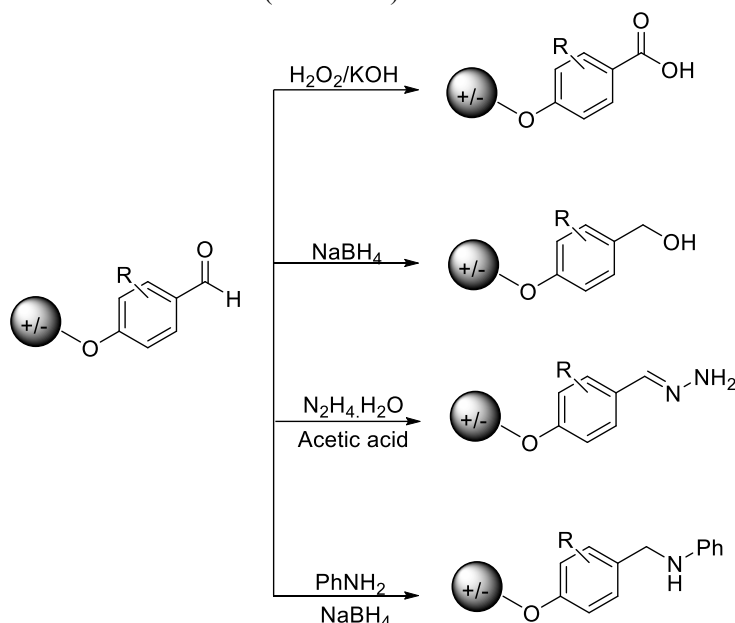


An alternative approach to preparation of ionic liquids

(Scheme 10)

5.7. Carboxyl-functionalized ionic liquids or benzylic alcohol-functionalized ionic liquids

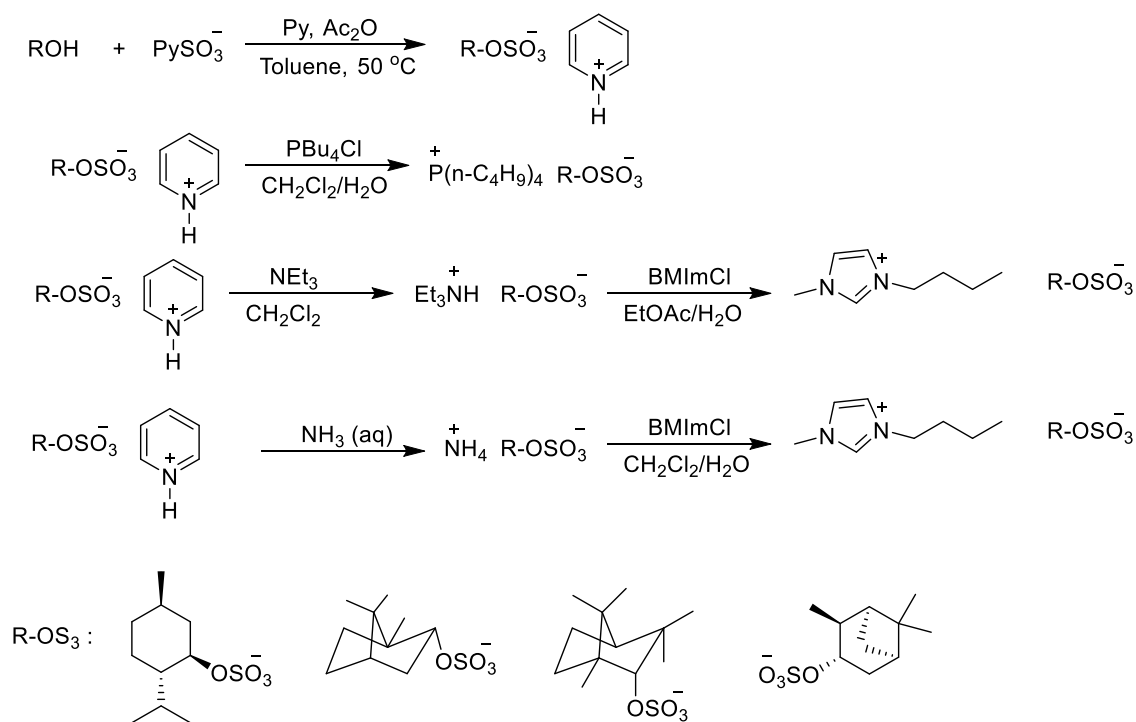
The aldehyde-functionalized ionic liquids can be employed as task-specific ionic liquids or as intermediates. They can be transformed into other functionalized ionic liquids thereby broadening a range of applications. These aldehyde-functionalized ionic liquids further form the corresponding carboxyl-functionalized ionic liquids or benzylic alcohol-functionalized ionic liquids. They were oxidised in the presence of $\text{H}_2\text{O}_2/\text{KOH}$ or reduced by NaBH_4 leading to the formation of products containing benzylic alcohol. Condensation of these functionalized ones with hydrazine hydrate and aniline were done under reductive amination conditions (Scheme 11).^{xxv}



(Scheme 11)

5.8. Chiral sulphate anions based Ionic Liquid

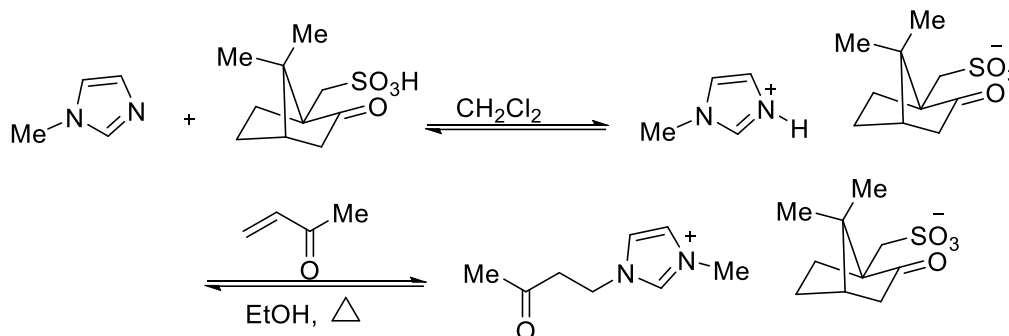
Alcohols were used to create chiral sulphate anions in high quantities. Tetra(n-butyl)phosphonium and 1-butyl-3-methylimidazolium (BMIM) cations were produced through ion metathesis (Scheme 12).^{xxvi}



(Scheme 12)

5.9. Ionic liquids containing prochiral keto-functionalized cations

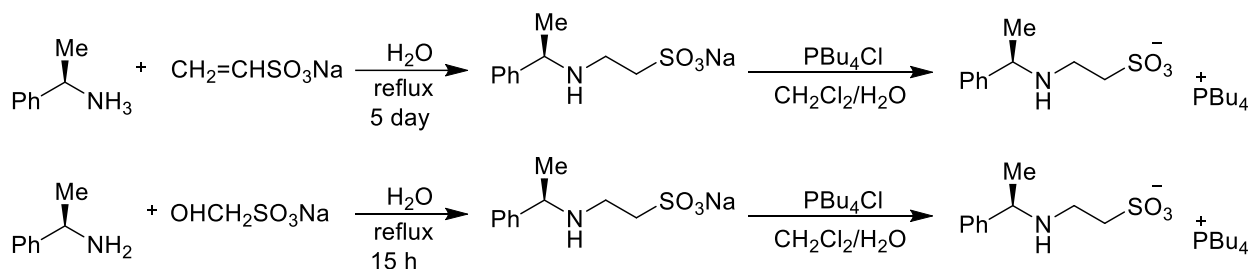
Prochiral keto-functionalized cations were hydrogenated in the presence of their chiral, enantiomerically pure counterion using ruthenium catalyst. [N-(30 oxobutyl)-N-methylimidazole] [(C)-camphorsulfonate] was created as an illustration of an *N, N'*-dialkylated, prochiral imidazolium salt (Scheme 13).¹⁷



(Scheme 13)

5.10. Chiral ionic liquids with the chiral sulfonate anions

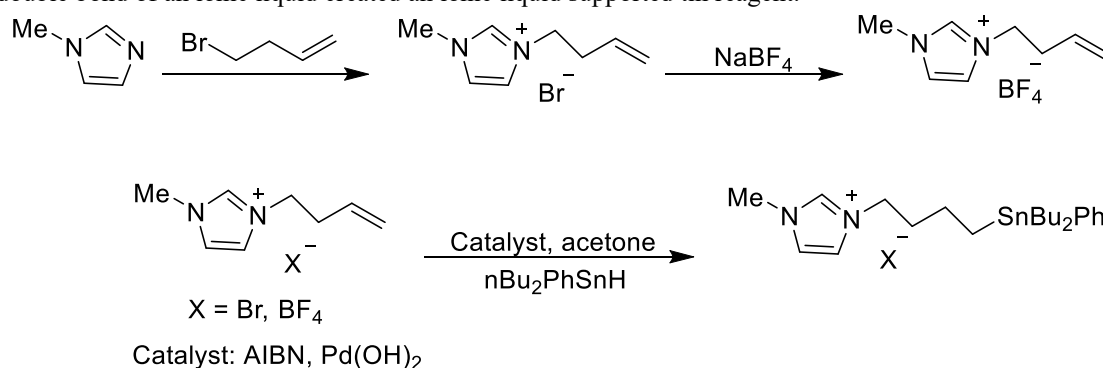
(*R*)-phenylethylamine and the bisulfite adduct of formaldehyde, or sodium vinylsulfonate were used to synthesize Brønsted basic chiral ionic liquids with the chiral sulfonate anions (Scheme 14).^{xxx}



(Scheme 14)

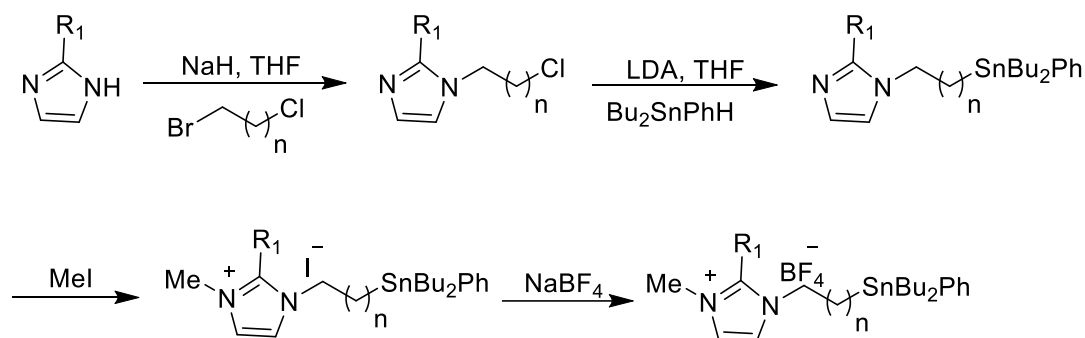
5.11. Ionic liquid supported tin reagents

a) The different methods of synthesis of IL-supported tin reagents are shown in (Schemes 15). Hydrostannylation of the terminal double bond of an ionic liquid created an ionic liquid supported tin reagent.



(Scheme 15)

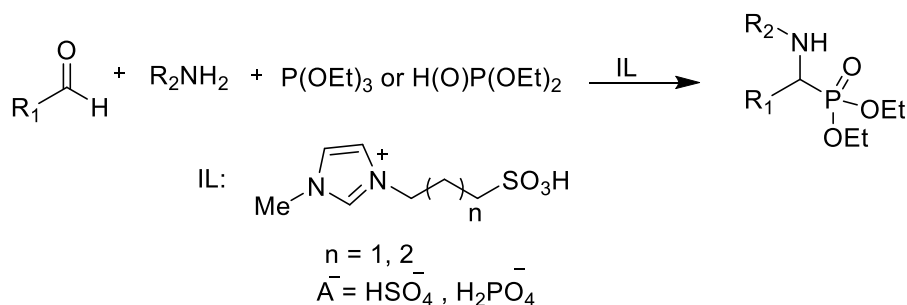
b) Ionic liquid supported tin reagents were synthesized using tin reagents and imidazoles containing halogen atoms in their alkyl chains, followed by the formation of the ionic liquid by simple addition of methyl iodide (Scheme 16).^{xxvii}



(Scheme 16)

5.12. Halogen-free Sulfonic acid-functionalized IL:

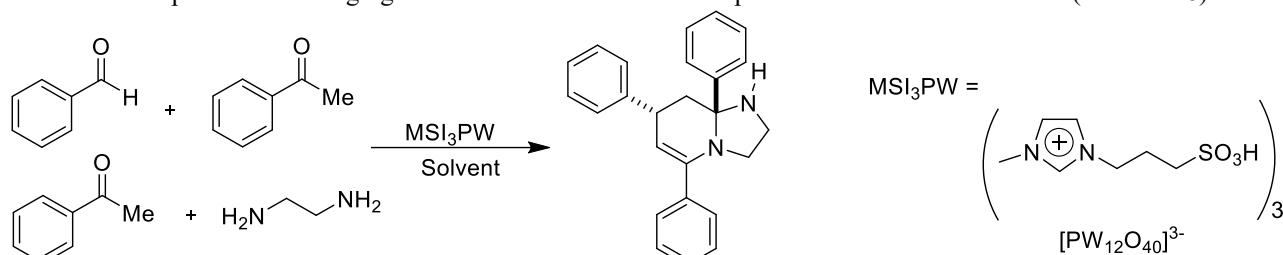
Synthesis of α -aminophosphonates was performed using Sulfonic acid-functionalized halogen-free imidazolium-based ionic liquids. The process involves efficient and recyclable catalyst at room temperature and the overall reaction comes under one-pot three-component condensation reaction in aqueous media. Products were obtained in good yields (Scheme 17).^{xxviii}



(Scheme 17)

5.13. Heteropolyacid-Containing Ionic Liquid-Catalyzed Multicomponent Synthesis of Bridgehead Nitrogen Heterocycles: Mechanisms and Mitochondrial Staining

An effective and catalytic MCR process was created to produce hexahydroimidazo[1,2-a]pyridine derivatives. A task-specific ionic liquid using heteropolyacids as the catalyst enabled synthesis of 10 novel derivatives all in the trans configuration. The use of the elegant charge tag strategy enabled a catalytic cycle to be developed for this transformation. The incorporation of coumarin scaffolds in the structure made the dye prone to be used as a live-cell imaging probe. The use of the new compound in bioimaging showed the MCR adduct had a preference for mitochondria.^{xxix}(Scheme 18)



Model Reaction for the Synthesis of HImP Catalyzed by MSI₃PW
(Scheme 18)

VI. CONCLUSION

In conclusion, synthesis of various ionic liquids are discussed along with varied pathways. Different ionic liquids prepared can be utilized in numerous organic transformations to produce complex structure. They are promising green materials with wide applications in material science. Their unique properties, such as low volatility, recyclability, and catalytic efficiency, make them excellent alternatives to conventional solvents. They can act as both solvents and catalysts, supporting sustainable organic synthesis. Functionalized ILs further expand their usefulness. Immobilizing ILs on solid

supports helps overcome limitations, combining stability with efficiency. Overall, ionic liquids represent eco-friendly, versatile, and efficient tools for modern chemical research.

ACKNOWLEDGEMENTS

Research Scholar Mr. Natwar Kumar Jha is grateful to PG Dept of Chemistry, M R M College, Darbhanga India for providing necessary help in constructing the article in current format.

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