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SYNTHESIS OF NEW PHASE TRANSFER CATALYST AND ITS APPLICATIONS

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Abstract :

A mixture of α-phenyl ethyl amine (**A**), formic acid and formaldehyde on reflux to give N,N-dimethyl-α-phenylethylamine (**B**). This N,N-dimethyl-α-phenylethylamine reflux with n-butyl bromide to yield *N*-butyl-*N*,*N*-dimethyl-α-phenylethylammonium bromide (C). This catalyst is used in esterification reactions of esfenvalarate and cypermethrin, oxidation of aryl carbinols and alkylation of phenols.

Key Words :

α-phenyl ethyl amine, formic acid, formaldehyde, N,N-dimethyl-α-phenylethylamine.

Introduction :

A survey of literature reveals that very little work has been carried out on synthesis of phase transfer catalyst. A phase-transfer catalyst enables the reaction in a heterogeneous system between general organic compounds soluble in organic solvents and compounds soluble in water such as inorganic salts. The reaction can be accomplished in a biphasic system of an inexpensive nonpolar aprotic solvent and water without using DMSO or DMF which are high-polar solvents. A phase-transfer catalyst is soluble in both solvents, and it carries anions of inorganic salts into organic solvents and returns them into the water phase. Reactions usually progress under mild conditions with easy work-up procedures. For this reason, they are also used industrially. Typical phase-transfer catalysts are quaternary ammonium salts, crown ethers, and phosphonium compounds[1-4].

Since its introduction around 1965, phase transfer catalysis (PTC) has become a firmly established technique in synthetic organic chemistry[5]. Many reviews on synthetic methods using phase transfer catalysts have been published (Brandstrom, 1977; Dehmlow and Dehmlow, 1993; Weber & Gokel, 1977; Starks, 1994; Freedman, 1986). One of the major concerns in using a phase transfer catalyst (PTC) in soluble form is its separation from the reaction mixture. For efficient use of the catalyst and to meet product purity requirements, synthetic techniques using PTCs involve an additional separation for catalyst isolation and product purification [6-8].

Principal of Phase Transfer catalysis :

The principle of phase transfer catalysis (PTC) is brought forth well by Reuben and Sjoberg (1981). The principle of PTC is based on the ability of certain phase-transfer agents (the PT catalysts) to facilitate the transport of one reagent from one phase into another (immiscible) phase wherein the other reagent exists. Thus, reaction is made possible by bringing together the reagents which are originally in different phases. However, it is also necessary that the transferred species is in an active state for effective PT catalytic action, and that it is regenerated during the organic reaction[9].

Mechanism of PTC :

The mechanism of PTC reaction was first proposed in 1971 [10]. According to Starksí original work, a quaternary ammonium halide dissolved in the aqueous phase undergoes anion exchange with the anion of the reactant dissolved in the aqueous solution. The ion-pair formed can cross the liquid-liquid interface due to its lipophilic nature and diffuses from the interface into the organic phase. In the organic phase, the anion of the ion-pair being quite nucleophilic undergoes a nucleophilic substitution reaction with the organic reagent forming the desired product. The catalyst subsequently returns to the aqueous phase and the cycle continues. A prerequisite for a substance to function as a PT-Catalyst is to form ion-pairs soluble in the organic phase and to be transferred in a highly active state[11,12]. Other mechanisms that can be considered as phase-transfer catalytic are :

1. PTC of uncharged species: complexation and transfer of uncharged protic species or metal salts into the organic medium as complexes of the phase -transfer agent[13].
2. Electron-transfer catalysis for Redox systems[14-17].
3. Metal ion-transfer from aqueous solutions into water immiscible ionic liquids containing neutral complexing agents[18].

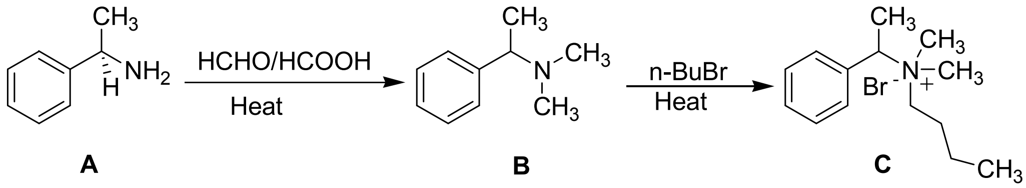
Experimental Section :

All melting points were determined in open capillary tube and were uncorrected. IR spectra were recorded with potassium bromide pellets technique, ^{1}H NMR spectra were recorded on AVANCE 300 MHz Spectrometer in DMSO using TMS as internal standard. Mass spectra were recorded on a FT VG-7070 H Mass Spectrometer using EI technique at 70 eV. All the reactions were monitored by Thin layer chromatography.

Material and Methods :

Preparation of new phase transfer catalyst *N*-butyl-*N*,*N*-dimethyl-α-phenylethyl ammonium bromide (C).

The compound *N*-butyl-*N*,*N*-dimethyl-α-phenylethyl ammonium bromide (**C**) was prepared starting from α-phenyl ethyl amine (**A**) by using formic acid and formaldehyde to get N,N-dimethyl- α-phenylethylamine (**B**) followed by reaction with n-butyl bromide (Scheme **1**).



(Scheme-1)

Results and Discussion :

Many of the industrially and economically important organic chemical intermediates and biologically significant compounds require in their production or process the use of high yielding procedures with little or no pollution. Phase transfer catalysis permits or accelerates reactions between ionic compounds and water insoluble organic substrates by transferring the ionic compounds into organic medium in the form of ionic pairs.

A need arose to search for developing a new and simple quaternary ammonium salt, such that the same may be used as a phase transfer catalyst in the ongoing some of the process development work under taken at different chemistry department in the world. In view of this a simple N-butyl-N,N-dimethyl-α-phenylethyl ammonium bromide catalyst has been prepared and its utility as phase transfer catalyst in various organic transformations is exploited and the results are reported.

Conclusion:

The synthesis of new new phase transfer catalyst and its utilization in various organic reactions is reported in this research which is used for many organic synthesis. This catalyst is used in esterification reactions of esfenvalarate and cypermethrin, oxidation of aryl carbinols and alkylation of phenols.

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