

Phase transfer catalysis in the chemistry of five-membered nitrogen-containing heterocycles

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The methods for synthesis and reactions of five-membered nitrogen-containing heterocycles under the conditions of phase transfer catalysis as well as the application of quaternized heterocycles as phase transfer catalysts are discussed.

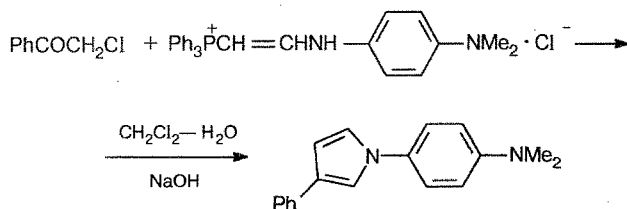
Key words: phase transfer catalysis, phase transfer catalysts, five-membered nitrogen-containing heterocycles, quaternized nitrogen-containing heterocycles.

The considerable advances of the last 10–15 years in the chemistry of nitrogen-containing heterocyclic compounds are closely related to the application of phase transfer catalysis (PTC) methods. The information published on this topic until the mid-eighties is generalized in reviews,^{1–3} monographs,^{4,5} and Keller's comprehensive compendium on PTC.^{6–8}

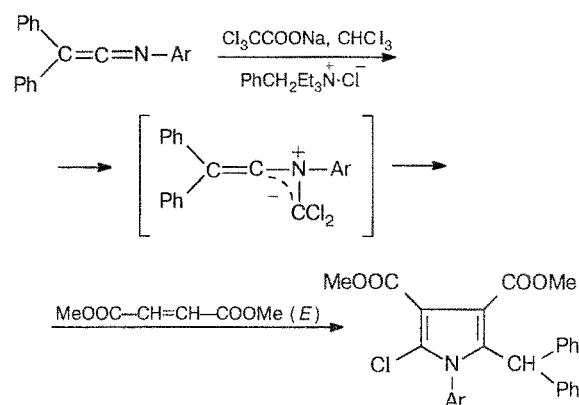
New data obtained mainly during the most recent 8–10 years on the application of the PTC method in the chemistry of five-membered heterocycles with one, two, three, and four nitrogen atoms in a ring are presented herein. Special attention is given to the problem of the application of quaternized nitrogen-containing heterocycles as phase transfer catalysts. Earlier this problem was not discussed at all.

Preparation of five-membered nitrogen-containing heterocycles

Several methods of constructing the pyrrole ring that are usually applied in the preparation of pyrroles under PTC conditions are known. First of all there is the Wittig reaction.⁹

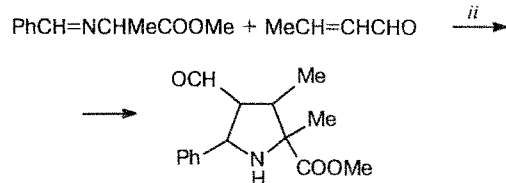
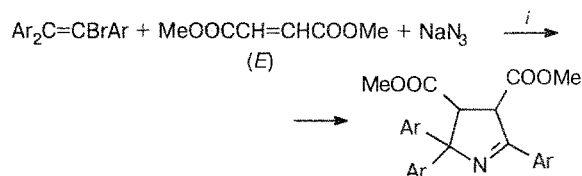


In the later work, substituted pyrroles were obtained by the reaction of dimethyl fumarate with ketenimine ylides formed in the reaction of dichlorocarbene with *N*-(2,2-diphenylvinylidene)anilines under PTC-generated conditions.¹⁰



It seems that the application of the PTC method would be very efficient in alkylation of enamine in the Hantzsch synthesis of pyrroles.

The advantage of PTC can be clearly seen in preparing functionally substituted pyrrolines and pyrrolidines, which cannot be synthesized by the direct reduction of the corresponding pyrroles (or by cyclization reactions).^{11,12}

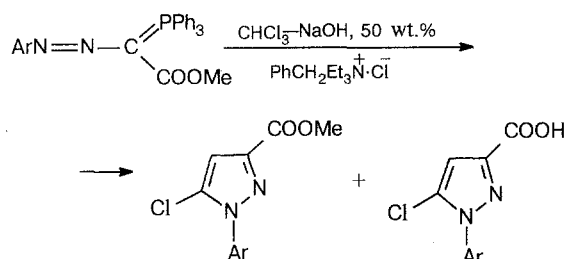


i. $h\nu$, $\text{CH}_2\text{Cl}_2-\text{H}_2\text{O}$, $\text{Bu}_4\text{N}^+\text{Cl}^-$
ii. K_2CO_3 , CH_3CN , $\text{PhCH}_2\text{Et}_3\text{N}^+\text{Cl}^-$

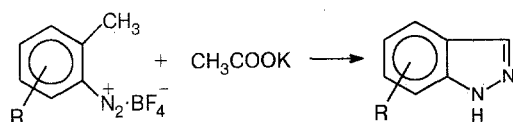
A convenient method for preparing *N*-cyano-pyrrolidine by alkylation of cyanamide with 1,4-dibromobutane in an aqueous solution of sodium hydroxide in the presence of Aliquat 336¹³ should be also mentioned.

The preparation of indoles and isoindolines under the PTC conditions is described in Refs. 13 and 14.

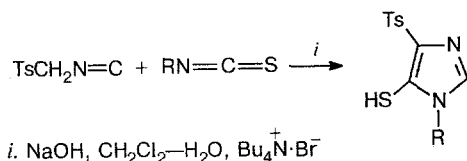
Several sufficiently simple and efficient methods for the syntheses of pyrazoles and pyrazolines under PTC conditions have been developed recently. One of them is the reaction of *gem*-dichlorocyclopropanol with hydrazine or phenylhydrazine in the presence of dibenzo-18-crown-6,¹⁵ and the second method is the reaction of aryl-azomethylenetriphenylphosphoranes with PTC-generated dichlorocarbene.¹⁶



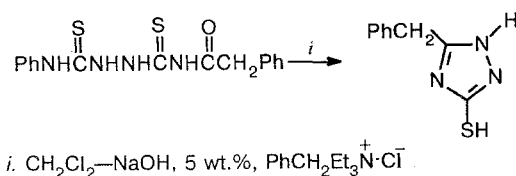
A peculiar method for preparing indazoles by the reaction between *o*-methylphenyldiazonium tetrafluoroborate and potassium acetate in the presence of 18-crown-6 was suggested in Ref. 17. The cyclization occurs at room temperature with a high rate to give indazoles in a 60–90 % yield.



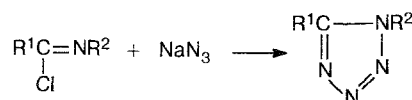
Finally, one of the few methods for the synthesis of 4,5-disubstituted imidazoles under PTC conditions, *i.e.*, dipolar cycloaddition of isocyanates to isonitriles, should be mentioned.¹⁸



Only two examples of preparing triazoles under PTC conditions are described.^{19,20} One of them is the following:



At the same time, this method becomes more and more popular for the tetrazole series.^{21–24} It is shown that the reactions of imidoyl chlorides with sodium azide in a methylene dichloride–water system in the presence of tetrabutylammonium bromide or 2,3-diphenyl-5-butyltetrazolyl bromide result in the formation of 1,5-disubstituted tetrazoles in 85–92 % yield. The reaction occurs at room temperature and is completed in 2 h after mixing the reagents.



On going from the liquid–liquid system to the solid–liquid system, tetrazoles are formed in the same high yield; however, the duration of the reaction increases to 12–14 h.

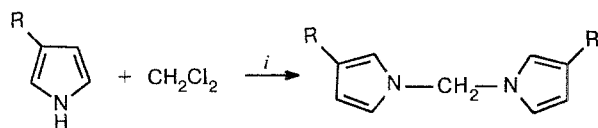
It should be emphasized that this method for preparing 1,5-disubstituted tetrazoles has indisputable advantages compared to the conventional one, *i.e.*, the reaction of imidoyl chlorides with extremely dangerous solutions of hydrazoic acid.²⁵ The preparation of tetrazolium salts by the oxidation of 1,3,5-trisubstituted formazans under PTC conditions is described in Refs. 22, 23, and 26.

Reactions of five-membered nitrogen-containing heterocycles

The study of chemical transformations of five-membered nitrogen-containing heterocycles under PTC conditions made it possible to substantially enlarge the synthetic possibilities of known reactions, develop simple methods for preparing compounds that were not easily accessible earlier, and consider from a new viewpoint many chemical processes whose mechanisms were previously considered "finally" established. Of the many reactions of heterocyclic compounds occurring under PTC conditions, alkylation, acylation, sulfonation, and silylation are studied better than others, while reactions with dihalocarbenes and redox reactions are studied to a lesser extent.

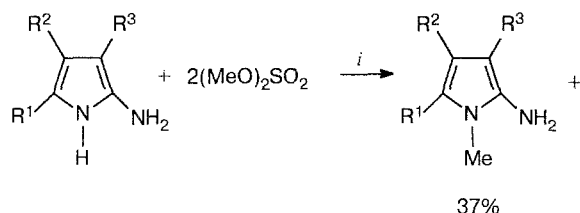
Any nitrogen-containing heterocycles that are able to form sufficiently stable heteroanions under the action of strong bases readily react with alkylating, acylating, sulfonating, and silylating agents. Quaternary ammonium salts and crown ethers are most often used in these processes as phase transfer catalysts. Depending on the properties of reagents, the reactions are carried out in the liquid–liquid or solid–liquid system.

Pyrrole and indoles in the liquid–liquid system are readily alkylated by alkyl halides and alkyl sulfates.^{27–29} The alkylation of pyrroles by methylene dichloride results in the formation of the corresponding bisderivatives in a high yield.³⁰



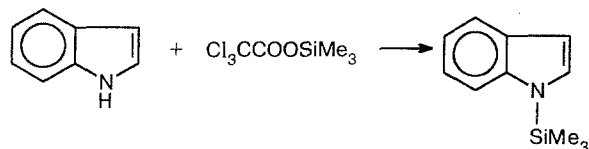
i. NaOH, 50 wt.%, $\text{Bu}_4\text{N}^+\text{I}^-$

The selectivity of the alkylation of 2-aminopyrroles by dimethyl sulfate depends on a ratio of the reagents.³¹ When the pyrrole : alkylating agent ratio is 1 : 1.1, the alkylation occurs only at the nitrogen atom of the ring. The reaction with a twofold excess of dimethyl sulfate results in the formation of all possible alkylation products.



i. CH_2Cl_2 —aqueous NaOH, $\text{PhCH}_2\text{Bu}_3\text{N}^+\text{Br}^-$

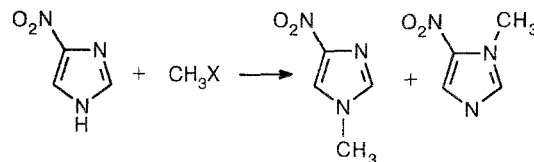
Acylation, sulfonation, and silylation of pyrrole and indole are also easy.^{32–34} Of the studies devoted to these reactions, Ref. 34 should be mentioned in which trimethylsilyl trichloroacetate is used as a silylating agent. The reaction occurs at 100 °C in the presence of potassium carbonate and with 18-crown-6 as the catalyst.



Pyrazoles and imidazoles are the most studied of all five-membered nitrogen-containing heterocycles in PTC. Many examples of alkylation of pyrazoles and imidazoles by alkyl sulfates³⁵ and functionally substituted alkyl halides,^{36,37} including 1,2-dichloro- and 1,2-dibromoethane³⁸ and alkyl carbonates,³⁹ are described.

Very interesting data have been obtained recently in studying the dependence of the selectivity of alkylation of 4-nitroimidazole with methyl iodide and dimethyl sulfate on the nature of the phase transfer catalyst.⁴⁰

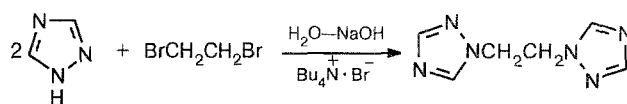
Quaternary ammonium salts and benzo-15-crown-5 were used as catalysts.



It was shown that 1-methyl-4-nitroimidazole is the main reaction product for any alkylating agents and phase transfer catalysts. At the same time, on going from methyl iodide to dimethyl sulfate, the fraction of 3-methyl-substituted derivative in the reaction products somewhat increases, but does not exceed, however, 32 %. Of all catalysts studied in alkylation by methyl iodide, tetrabutylammonium iodide most of all facilitated the formation of 3-methyl-4-nitroimidazole; and Aliquat 336 is the catalyst for alkylation by dimethyl sulfate. At the same time, it is noteworthy that 1-benzyl-2-methyl-4-nitroimidazole is the only reaction product, when 2-methyl-4-nitroimidazole is alkylated by benzyl chloride in the solid—liquid system in the presence of tetrabutylammonium bromide.⁴¹

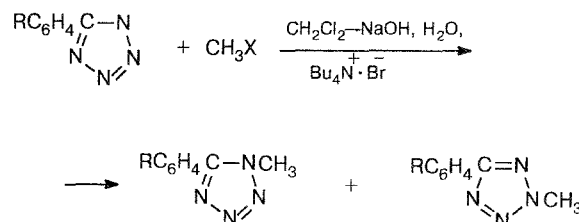
The data on acylation, sulfonation, and silylation of imidazole and benzimidazole are presented in Refs. 34 and 42.

Publications devoted to studies of the alkylation of 1,2,4-triazole and benzotriazole under PTC conditions have appeared recently. The most interesting results of synthesis are the alkylation of these compounds with dibromoethane.³⁸ Thus, bistriazolyethanes that were not easily accessible previously can be obtained in good yields.



The alkylation of 1,2,4-triazole and benzotriazole with 1-chloro-2-(pyrazol-1-yl)ethane is also described in Ref. 38.

The application of PTC in the chemistry of tetrazoles is illustrated by the example of such reactions as alkylation, acylation, and imidoylation.^{24,25} The alkylation of a large series of 5-aryltetrazoles with methyl iodide and dimethyl sulfide was studied.



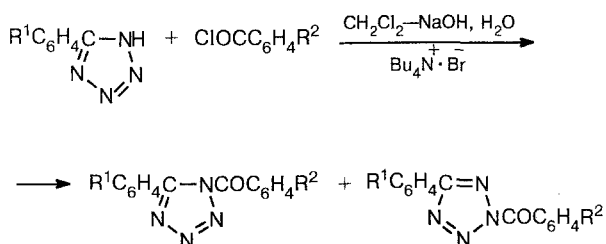
It was supposed that the application of PTC would result in a substantial change in the selectivity of the process.

However, it turned out that the ratio between N(1)- and N(2)-isomers formed in the alkylation of tetrazoles in the noncatalytic process is almost the same as that under PTC conditions. It has been established that the reason for this is the specific features of structures of tetrazole salts in which the tetrabutylammonium cation is arranged above the plane of the tetrazole ring. For this structure, the attack of an alkylating agent occurs from the side of the plane of the tetrazole cycle, which is opposite to that where the cation is located. This means that steric hindrances caused by a large size of the tetrabutylammonium cation should not affect the selectivity of alkylation.

The alkylation of tetrazole by 1,2-dibromoethane and 1-chloro-2-(pyrazol-1-yl)ethane was studied in Ref. 38. The authors of this study do not discuss the selectivity of alkylation, but, based on the data of ^1H and ^{13}C NMR spectra, believe that 1,2-bis(tetrazol-2-yl)ethane and 1-(pyrazol-1-yl)-2-(tetrazol-2-yl)ethane, respectively, are formed in this case.

It is of interest that no change in the selectivity of the reaction is observed in the alkylation of (1-phenyltetrazol-5-yl)thione under PTC conditions compared to that of the noncatalytic process. Only products of S-alkylation are formed in all cases.⁴³

It has been shown for the acylation of 5-aryltetrazoles by substituted benzoyl chlorides that two isomers, 1-acyl- and 2-acyltetrazoles, are formed in this process, not solely the 2-isomer supposed previously.

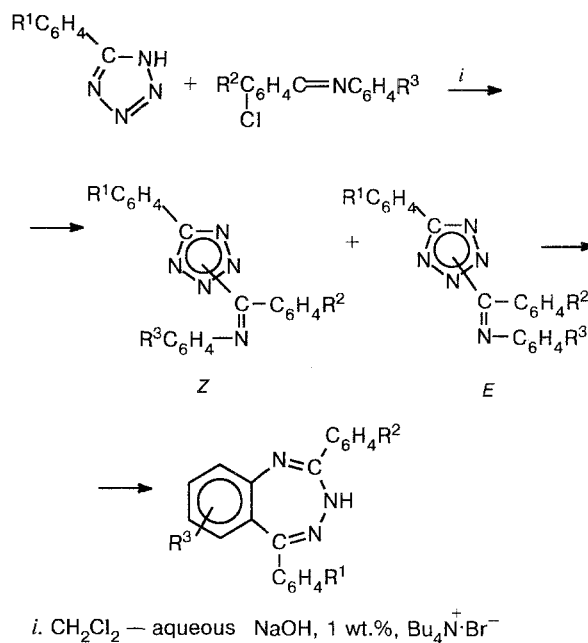


Finally, basically important results obtained for imidoxylation of 5-aryltetrazoles should be mentioned. The application of the PTC method to this reaction made it possible to correct substantially the mechanism of imidoxylation of 5-substituted tetrazoles and provided quite new unexpected possibilities in syntheses of heterocyclic compounds that were not easily accessible previously.⁴⁴⁻⁴⁶ It is established that the reactions between 5-aryltetrazoles and *N*-arylbenzimidoyl chlorides result in the formation of 1-imidoxy- and 2-imidoxy-5-aryltetrazoles that exist as *Z*- and *E*-isomers. The thermolysis of 1-imidoxy- and 2-imidoxy-5-aryltetrazoles results in 3*H*-1,3,4-benzotriazepines (Scheme 1).

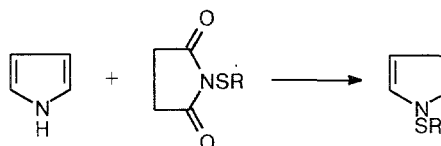
Of other reactions of five-membered nitrogen-containing heterocycles studied recently, the formation of *N*-alkylthiosubstituted derivatives of heterocycles, addition to dichloroacetylene, arylation, oxidation, and nitration should be mentioned first.

1-(Alkylthio)pyrroles in 75–95 % yields are formed in the reaction between pyrrole and *N*-(alkylthio)suc-

Scheme 1

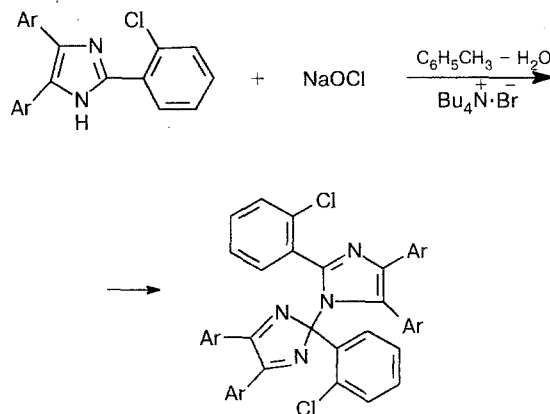


cinimide or (alkylthio)phthalimide in the methylene dichloride–water system in the presence of tetrabutylphosphonium bromide.⁴⁷

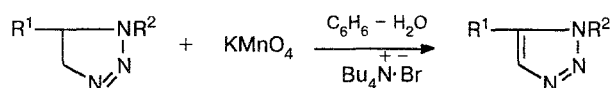


The corresponding 1-(alkylthio)indoles were obtained from indole under the same conditions. It is evident that the reaction has a general character and can be extended to other nitrogen-containing heterocycles. It was shown that such heterocycles as pyrazole and imidazole add to dichloroacetylene under PTC conditions,⁴⁸ and pyrazole is readily arylated by *p*-nitrofluorobenzene.⁴⁹

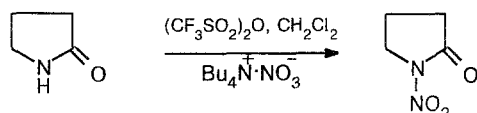
A unique case of oxidative dimerization has recently been found, when 2,4,5-trisubstituted imidazoles were treated with sodium hypochlorite.⁵⁰



It was mentioned that 1,2,3-triazoles are oxidized by potassium permanganate under PTC conditions to form the corresponding 1,2,3-triazoles.⁵¹



The only example of direct PTC nitration has recently been presented in Ref. 52.



One may believe that this method for the introduction of the nitro group will be extremely efficient for nitration of substrates sensitive toward acids.

Quaternized nitrogen-containing heterocycles as phase transfer catalysts

The interest in nitrogen-containing heterocycles quaternized at the nitrogen atom as phase transfer catalysts is associated with the fact that the usually used for these purposes ammonium, arsonium, and phosphonium salts are highly toxic, insufficiently stable at high temperatures, sometimes incapable of being regenerated, and in several cases not easily accessible. In recent years, several works on the application of quaternized nitrogen-containing heterocycles as phase transfer catalysts were published, but only tetrazolium²³ and pyridinium^{53–55} salts were systematically studied.

It is shown that pyrazolium salts are efficient phase transfer catalysts in denitration of trinitroanilines.⁵⁶ *N*-Alkylthiazolium bromides and perchlorates were successfully used in benzoin condensation.^{57,58} Unlike tetrabutylammonium cyanide that catalyzes only the condensation of aromatic aldehydes, *N*-alkylthiazolium salts can be applied in condensation reactions of both aromatic and aliphatic aldehydes.

The application of tetrazolium salts as phase transfer catalysts has first been described in 1985.⁵⁹ The catalytic activity of a large series of 2,3,5-trisubstituted tetrazolium salts was studied in Refs. 22 and 26. Extraction constants were calculated from the experimental data obtained in the study of distribution coefficients of these compounds, and it was shown that the catalytic activity of 2,3,5-triphenyltetrazolium bromide is not lower than that of such a widely applied phase transfer catalyst as tetrabutylammonium bromide.

It is established that the thermal stabilities of 2,3,5-trisubstituted tetrazolium salts are considerably greater than those of quaternary ammonium salts. The intense thermal decomposition of tetrazolium salts begins at 200–255 °C, depending on the nature of substituents in the heterocycle. Alkylation and acylation of hetero-

cycles,²⁴ formation of esters⁵⁹ and heterocycles,^{44–46} oxidation of imines to oxaziridines,⁶⁰ and oxidation of substituted toluenes to the corresponding benzoic acids^{26,61} are noteworthy of the reactions studied, using tetrazolium salts as phase transfer catalysts. The application of tetrazolium salts is most efficient in the reactions that occur in an organic solvent–water system with a neutral or acidic aqueous phase and in the cases when the catalyst should have a high thermal stability.

Many PTC reactions were studied using *N*-alkyl-4-(*N,N'*-dialkylamino)pyridinium salts.^{53,54,62} It is mentioned that the thermal stabilities of these compounds are considerably greater than that of tetrabutylammonium bromide, and they can easily be regenerated. Pyridinium salts can be applied very efficiently in aromatic nucleophilic substitution and dehydrohalogenation. Pyridyl sulfoxides and pyridine *N*-oxides also exhibit high catalytic activity in nucleophilic substitution.^{63,64} The advantages of these catalysts are their easy accessibility and possibility of complete recovery from reaction mixture.

The application of quaternized nitrogen-containing heterocycles as chiral phase transfer catalysts with sufficiently high selectivities is described in Refs. 4, 5, 65, and 66.

Finally, the publications about the application of five-membered nitrogen-containing heterocycles as co-catalysts in PTC reactions have appeared quite recently. It was shown that the oxidation of alkenes by sodium periodate in the presence of imidazole under PTC conditions results in the formation of the corresponding oxiranes in quantitative yields.⁶⁷

The discussion of the experimental material on the application of PTC in the chemistry of nitrogen-containing heterocycles shows a large flexibility of this method. At present, it is very difficult to conceive any reactions involving heteroanions, which could not be performed under PTC conditions. Here the advantages of this method are indisputable. Unfortunately, this does not concern yet the application of PTC to cationic reactions, although these processes are of great interest. Almost no attention is given to so-called "reverse phase transfer catalysis," when a substrate is transferred from an organic to aqueous phase where the reaction occurs.⁶⁴ There are only few works devoted to studying the mechanism of phase transfer catalytic reactions.

At the same time, the application of PTC in chemistry has already gone far beyond pure laboratory studies being, more and more often realized in industrial processes.^{53,68}

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