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THE REACTIVITY OF N-SUBSTITUTED AZAAROMATICS

In the present review dealing with the reactivity of N-substituted azaaromatics their reduction as well as reactions proceeding on ring carbon atoms and on nitrogen atom are described; also the reactivity of porphyrins and of complexes possessing these systems is reported.

N-substituted azaaromatics deserve an attention in view of their interesting reactivity [1—10] and biological activities [11—16]; they may serve as intercalators of nucleic acids, this fact being connected with the search for antineoplastic agents [17—19]. These compounds find application in redox systems [20—26] promising in the study of biomimetic processes [27, 28] and in solar energy conversion and storage [29, 30]; some among them may be used as dyes [31—34] and surfactants [35—38]. Polymers possessing such systems show interesting properties and inclusion complexes where N-substituted azaaromatics play the role of a host or of a guest may be used in molecular electronic devices [43—46].

N-substituted azaaromatics are widely applied in organic syntheses [47—50]; their cyclization reactions [51—55] and especially cycloaddition reactions of ylides generated from these salts [56—63] are convenient approaches to polycyclic systems, often difficult to obtain on other routes.

As cyclization reactions of N-substituted azaaromatics are a large topic, they are not included here.

The present paper, a continuation of our former reviews [64—67] and experimental papers [60—63, 68—70] deals with the reactivity of N-substituted azaaromatics.

There will be described:

1 — reduction of N-substituted azaaromatics,

2 — reactions of N-substituted azaaromatics proceeding on ring carbon atoms and on substituents at the latter,

3 — reactions of N-substituted azaaromatics proceeding on nitrogen atom and on substituents at the latter,

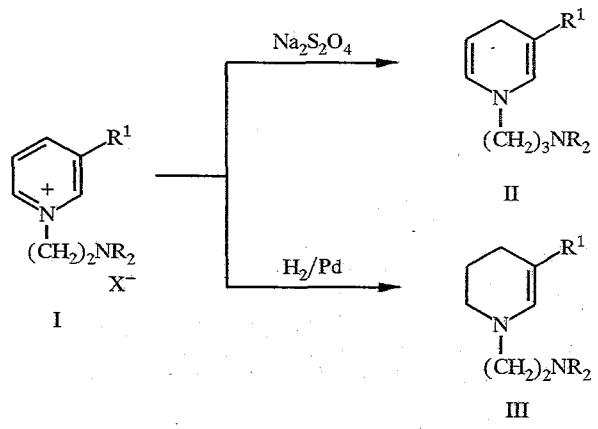
4 — reactions of porphyrins bearing N-substituted azaaromatic moieties,

5 — reactions of complexes of N-substituted azaaromatics.

1. REDUCTION OF N-SUBSTITUTED AZAAROMATICS

From numerous reduction reactions of N-substituted azaaromatics leading to dihydro- [47, 71—73], tetrahydro- [74, 75] or hexahydroderivatives [74—77] chosen examples will be described.

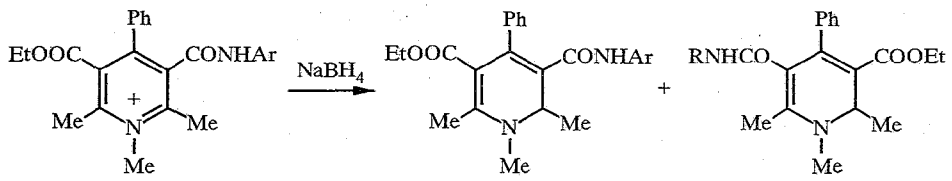
The results of the reduction of pyridinium salts (I) are strongly dependent on the nature of the reducing agent, e.g. the reaction of I with sodium dithionite affords 1,4-dihydropyridines (II) while the palladium catalyzed hydrogenation gives rise to tetrahydropyridines (III) [78].



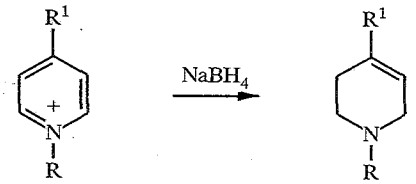
R = Me, Et; R^1 = CN, $CONH_2$, COMe, COOMe

X = Cl, Br

An example of the reduction of pyridinium salts yielding 1,2-dihydropyridines is the following reaction [79].



Salts IV reduced with sodium borohydride afford tetrahydropyridines V, useful in the synthesis of monoamine oxidase B inhibitors [80].

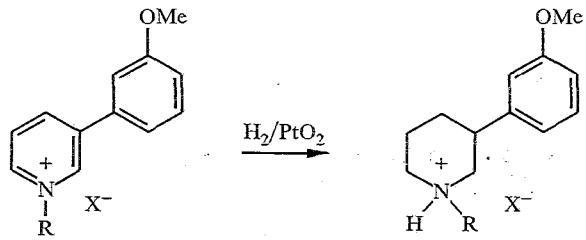


IV V

R = Me, $-CH_2-CH=CH_2$, $-CH_2-C\equiv CH$, Pr, cyclopentyl

R^1 = Ph, $-CH=CH_2$, $-CH=CH-Ph$

The reduction of salts VI with H_2/PtO_2 leads to piperidine derivatives VII [81].

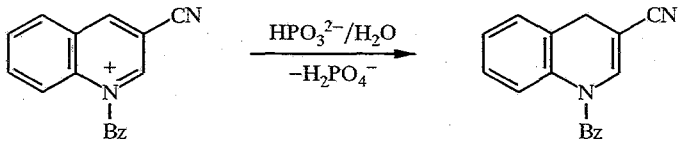


VI VII

R = Pr, Bz

X = Cl, Br

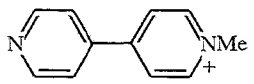
In the study of NAD^+ models reduction of 1-benzyl-3-cyanoquinolinium ion by phosphonate dianion was examined. The reaction was performed in aqueous propanol; the main product is 1,4-dihydroquinoline VIII with a trace of 1,2-isomer [73, 82].



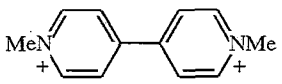
VIII

A considerable attention deserve viologens, compounds widely applied as mediators in catalytic photolysis of water and as redox indicators in biological systems [64, 83].

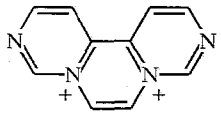
Among numerous reduction reactions of viologens [84—87] it should be mentioned the cathodic reduction of compounds IX—XII investigated by cyclic voltammetry and spectroelectrochemistry; the reduction of these quaternary salts is easier than in the case of corresponding free bases [88].



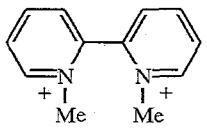
IX



X



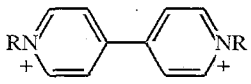
XI



XII

It was found that the reduction of dialkyl viologens by sodium thionite is influenced by the presence of polyelectrolytes, e.g. poly(styrenesulphonate). Polyelectrolytes stabilize the long-chain radical cations formed in the reaction.

The dimerization of dialkyl viologens XIII was investigated in the presence and in the absence of polyelectrolytes. The interactions leading to the association of the reduced species increase with the length of the alkyl chains, i.e. with the hydrophobicity of viologen molecules. The dimerization of reduced species derived from viologens of long alkyl chains decreases in the presence of polyelectrolytes [89—94].



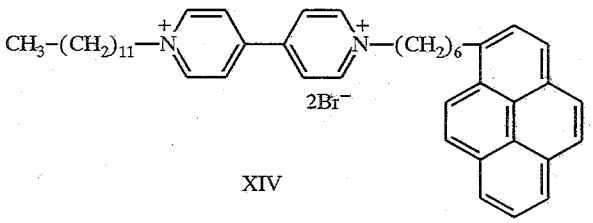
XIII

R = C_4H_9, C_7H_{15}, C_8H_{17}

In the study of solar energy conversion and biomimetic processes, intra and intermolecular photoinduced electron transfer reactions of bis-quaternary salt XIV solubilized in micellar solutions of a hexadecyltrimethylammonium chloride were examined [95].

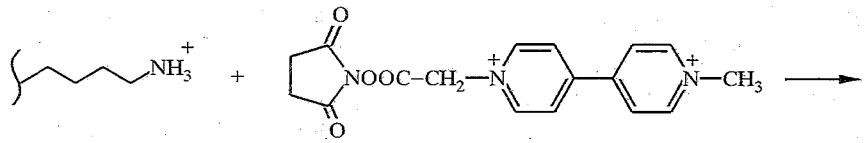
In XIV the viologen fragment acts as an electron acceptor and the pyrene one is a photosensitizer. The rate constants of the intramolecular electron-transfer

reactions are determined by molecular conformation of XIV in micelles, while the rate constants of the intermolecular photoinduced electron-transfer between 1-hexylpyrene and dodecylviologen are determined by the diffusion of XIV in a micelle [95].



XIV

The reaction of metmyoglobin XV with viologen XVI results in XVII.

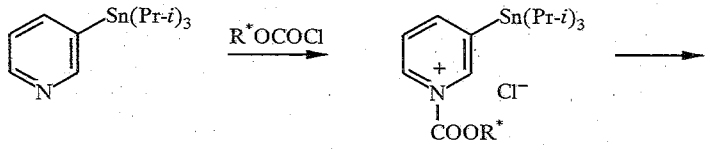


XV XVI

XVII

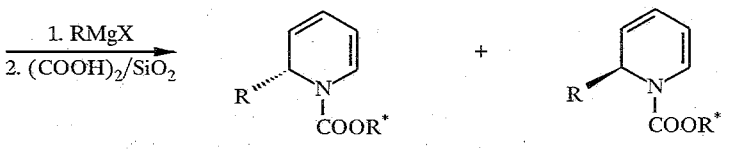
It was established that XVII undergoes a facile reduction with dithionite to give deoxymyoglobin, a Fe(II) form of myoglobin. The rate of the reduction is higher than that of native metmyoglobin; the reduction of the attached viologen is followed by the rapid electron transfer from the viologen radical cation to the heme iron center [96].

Examples of other reactions of N-substituted azaaromatics, leading to dihydroderivatives will be also presented here.



XXI XX

XXa R^* = (−)-8-(4-phenoxyphenyl)menthyl
XXb R^* = (−)-8-phenylmenthyl



XVIII XIX

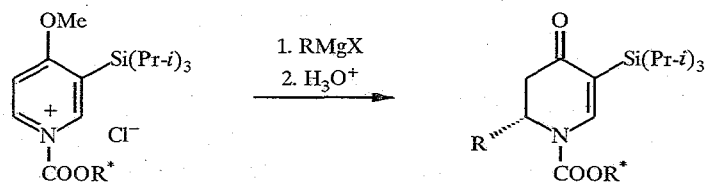
R = *p*-Me−C_6H_4, *c*-hexyl

Two asymmetric syntheses of 1-acyl-2-alkyl-1,2-dihydropyridines XVIII and XIX have been performed by the addition of Grignard reagents to chiral 1-acylpyridinium salts XX.

In the following process stannane XXI was used as the starting material, as the triisopropylstannyl group is bulky enough to block attack by the Grignard reagent at the C-2 and C-4 positions and may be easily removed with oxalic acid. Chiral 1-acylpyridinium salts XX were obtained in situ from XXI and the enantiopure chloroformate XXII.

Reaction of XX with a Grignard reagent and the subsequent treatment with silica gel containing oxalic acid yield chiral 1,2-dihydropyridines XVIII and XIX. The diastereoselectivity of the above reactions ranged from 76 to 92%.

In the next experiment the chiral pyridinium salt XXIII, prepared *in situ* from 4-methoxy-3-(triisopropylsilyl)pyridine and (−)-8-phenylmenthyl chloroformate was treated with Grignard reagent to afford dihydropyridone XXIV in high yield.

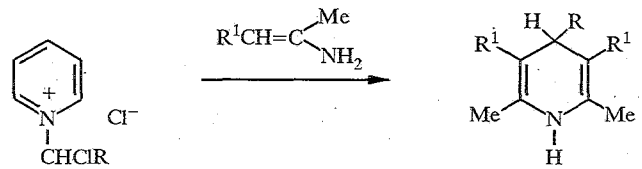


XXIII XXIV

R^* = (−)-8-phenylmenthyl

These studies are important for the enanthioselective synthesis of various alkaloids *via* chiral 1-acyl-1,2-dihydropyridine intermediates [97].

The modified Hantzsch reaction of pyridinium salts XXV with enaminocarbonyl derivatives provides N-unsubstituted 1,4-dihydropyridines XXVI [98].

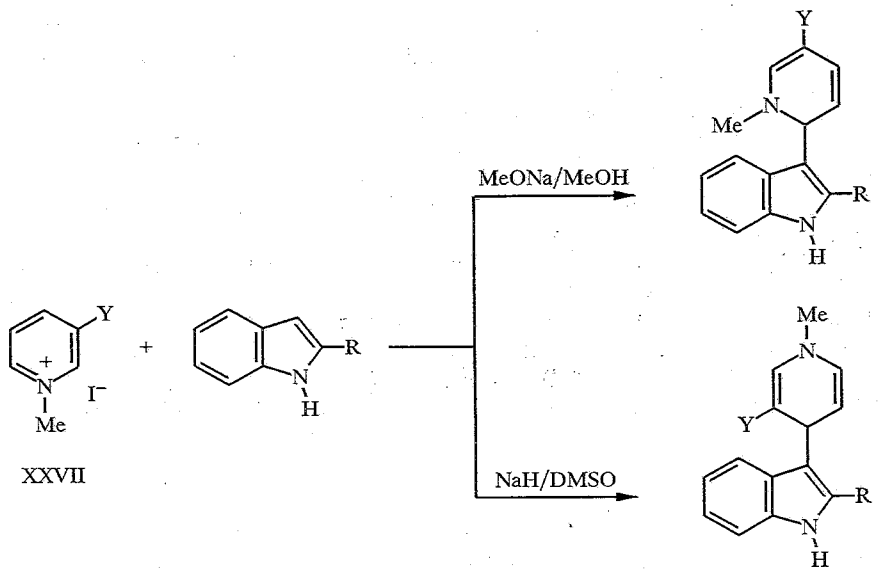


XXV XXVI

R^1 = CN, COMe, COOMe, COOBu-*t*

R = alkyl, Ph, 2-furyl, 2-thienyl

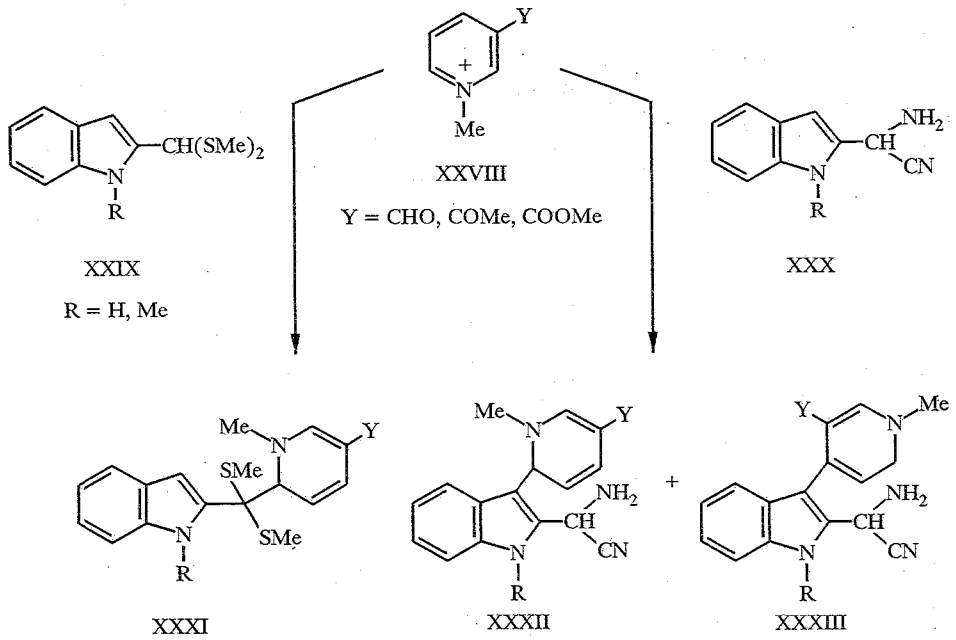
Pyridinium salts XXVII react with indoles to give 1,2- or 1,4-dihydropyridines, respectively, depending on the procedure used as shown below [99—101].



XXVII

Y = CHO, COMe, COOMe, –CH=CHCOOMe
R = H, Me

Reactions of pyridinium salts XXVIII with anions derived from dithioacetals XXIX and α-amino nitriles XXX give dithioketals XXXI or a mixture of dihydropyridylindoles XXXII and XXXIII respectively [102—105].



XXIX

R = H, Me

XXVIII

Y = CHO, COMe, COOMe

XXX

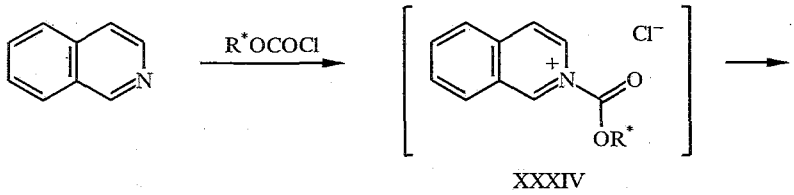
XXXI

XXXII

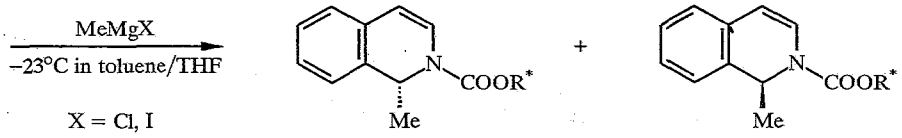
XXXIII

Compounds XXXI are interesting as precursors of 2-acylindole alkaloids, while XXXIII are promising as calcium antagonists.

The chiral N-acylisoquinolinium salt XXXIV obtained in situ by treatment of isoquinoline with (–)-8-phenylmenthyl chloroformate reacts with Grignard reagents to give a mixture of diastereoisomeric 1,2-dihydroisoquinolines XXXV and XXXVI [106].



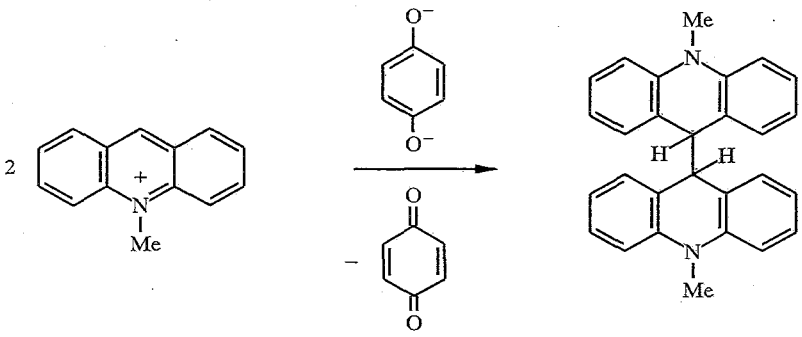
XXXIV



XXXV XXXVI

R^* = (−)-8-phenylmenthyl

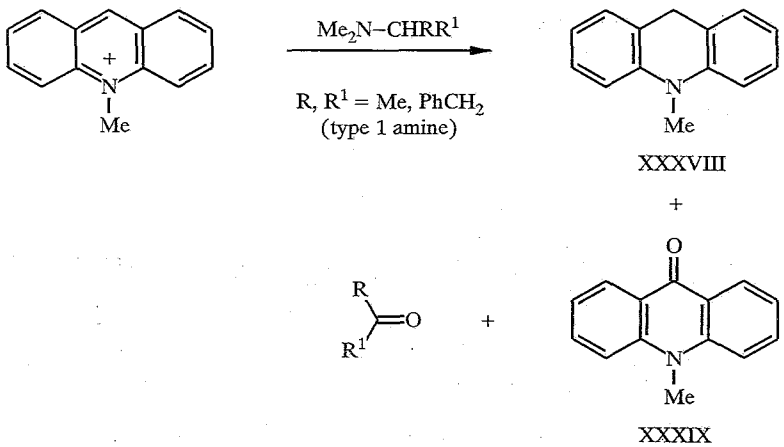
Investigating electron-transfer reactions it was observed that 10-methylacridinium ion treated with hydroquinone dianion yields the dimer XXXVII and *p*-benzoquinone [107, 108].



XXXVII

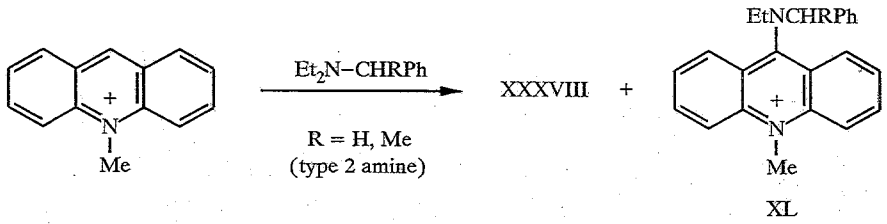
In the study of NAD^+/NADH system, the reaction of 10-methylacridinium perchlorate with a series of tertiary amines was investigated. The process depends strongly on the bulkiness of amines.

In the case of N,N-dimethylamines, referred to as the type 1 amines, i. e. Me_2NCHRR^1 (R, R^1 = H, $PhCH_2$), the products are 10-methyl-9,10-dihydroacridine XXXVIII, N-methylacridone XXXIX and ketone $RR^1C{=}O$.



XXXVIII XXXIX

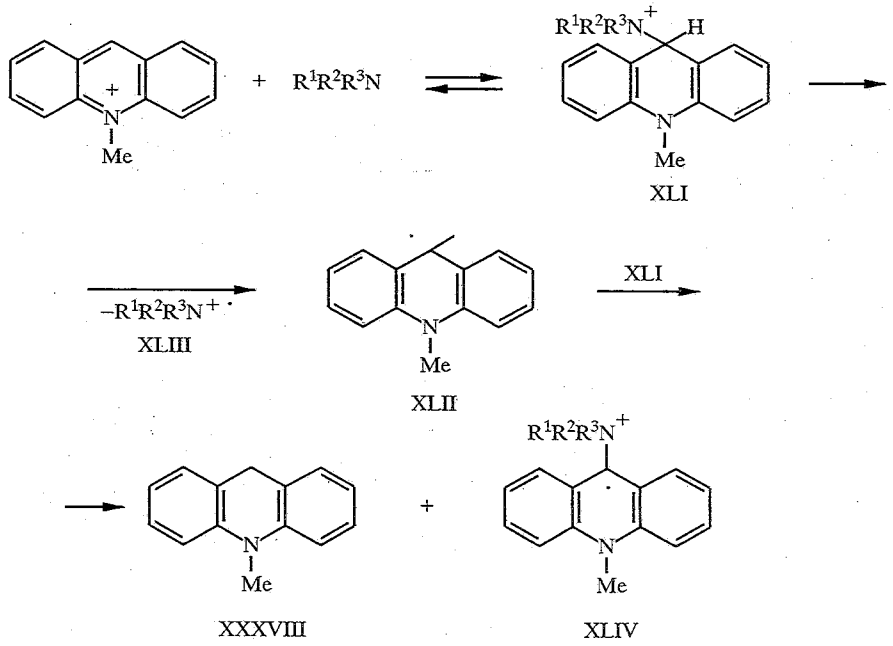
In the case of more bulky amines, referred to as the type 2 amines, i. e. $Et_2NCHRPh$ (R = H, Me), the reaction leads also to XXXVIII, along with salt XL.

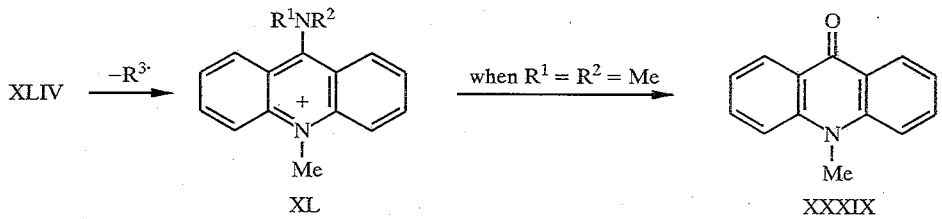


XL

The predominant mechanism involves the primary formation of the adduct XLI dissociating into the acridinyl radical XLII and an aminium cation radical XLIII.

The subsequent hydrogen-atom transfer from XLI to XLII affords XXXVIII and the radical cation XLIV, which releases one of the substituents as a free radical to give XL. For the type 2 amines XL is the end product, and for the type 1 amines it is hydrolyzed to the acridinone XXXIX [109, 110].

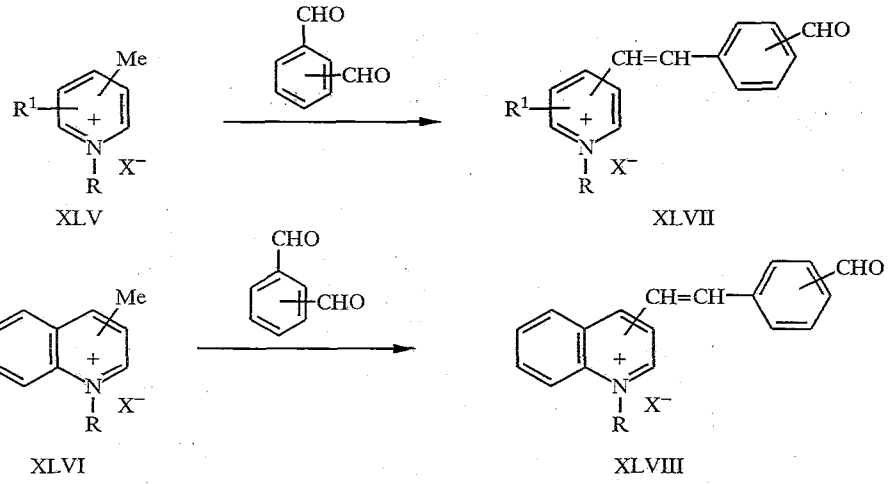




2. REACTIONS OF N-SUBSTITUTED AZAAROMATICS PROCEEDING ON RING CARBON ATOMS AND ON SUBSTITUENTS AT THE LATTER

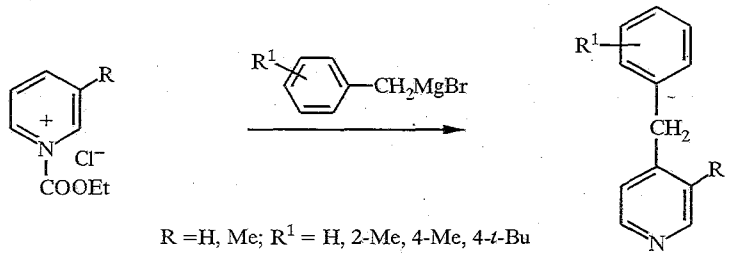
To reactions of N-substituted azaaromatics proceeding on ring carbon atoms belong processes leading to cyanine dyes [32—34]; chosen examples will be described here.

Treatment of pyridinium and quinolinium salts XLV and XLVI with phthalaldehydes affords photosensitive dyes XLVII and XLVIII, respectively [111, 112].



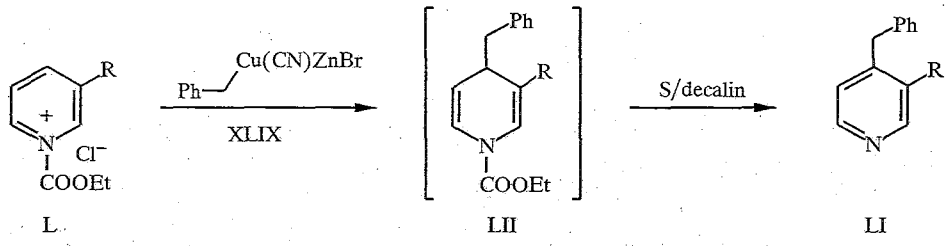
R = H, alkyl, aralkyl; R^1 = H, lower alkyl; X = anion

Among other reactions of N-substituted azaaromatics proceeding on ring carbon atoms the regiospecific addition of benzylic Grignard reagents to pyridinium salts should be mentioned [113].



R =H, Me; R^1 = H, 2-Me, 4-Me, 4-*t*-Bu

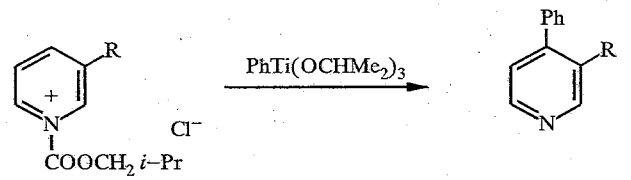
The addition of benzylic zinc cuprate XLIX to pyridinium salts L and the subsequent oxidation with sulfur lead to benzylpyridines LI. This highly regioselective reaction proceeds *via* the intermediate 1,4-dihydropyridine LII [114—118].



L LII LI

R = Me, Cl, Br, CHO, COOMe

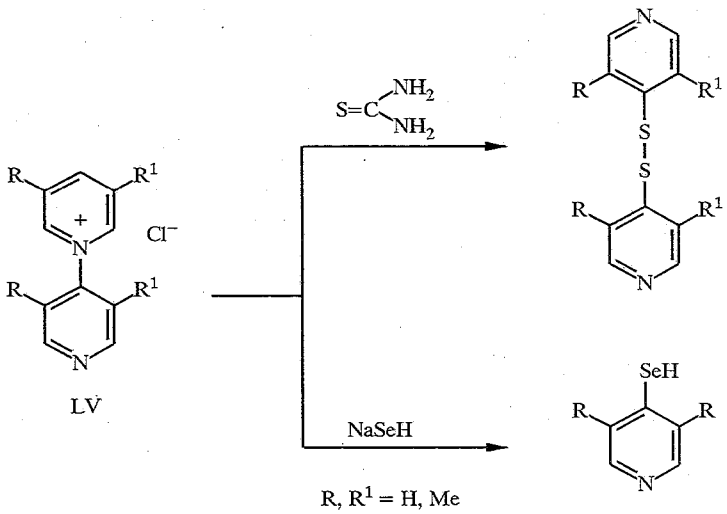
The arylation of the salts LIII with phenyltriisopropoxytitanium afford pyridines LIV in a completely regioselective manner [119].



LIII LIV

R = H, CN

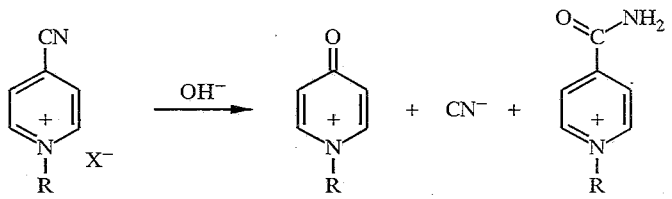
Following reactions of pyridylpyridinium chloride LV have been performed [120, 121].



LV

R, R^1 = H, Me

The alkaline hydrolysis of 4-cyanopyridinium halides LVI gives rise to two products, N-alkyl-4-pyridone LVII and N-alkyl-4-carbamoylpyridinium LVIII.

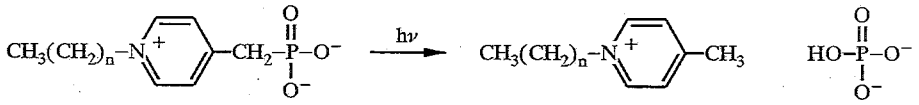


LVI LVII LVIII

R = n-C_6H_{17}, n-$C_{12}H_{25}$; X = Cl, Br, I

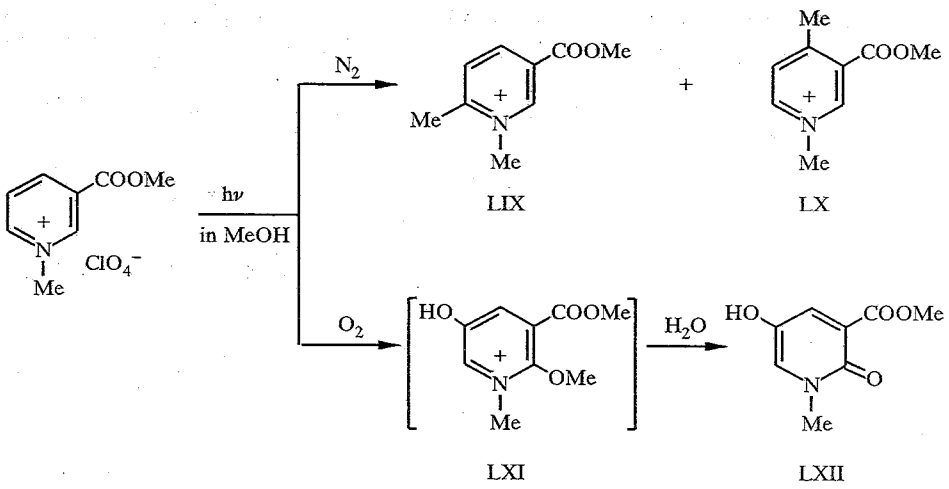
Micelles of cetyltrimethylammonium bromide and 3-[N,N,N-dimethyl-(dodecyl)ammonium]propane-1-sulphonate catalyse the hydrolysis of LVI and increase the ratio LVII/LVIII [122—128].

In the search for surface-active compounds, the irradiation of 1-alkyl-4-pyridiniomethylphosphonates bearing long alkyl chains in alkaline aqueous media was examined. The observed C—P bond cleavage results in 1-alkyl-4-methylpyridinium phosphates [129, 130].



n = 0, 7, 9, 11, 13, 15, 17

It was established that the photoreaction of 3-methoxycarbonyl-1-methylpyridinium perchlorate carried out in methanol under nitrogen affords 2- and 4-methyl substituted pyridinium salts LIX and LX, while in the presence of oxygen the product of the reaction is the unstable salt LXI, converted immediately into pyridone LXII. The mechanism of the methylation under nitrogen is proposed [131, 132].



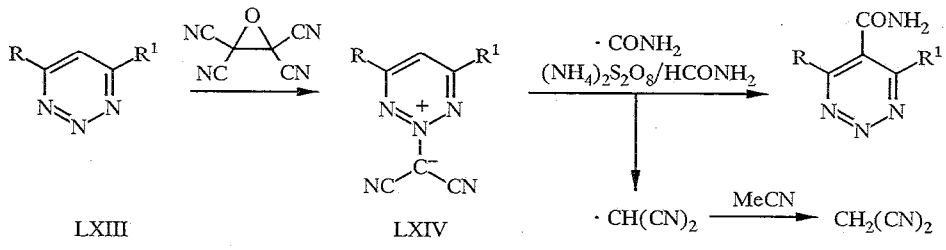
LIX LX

LXI LXII

The investigation of the H/D exchange in 1,2,4-trimethylpyridinium perchlorate has shown that the rate of isotopic exchange of 2-methyl group is higher than that of 6-methyl group [133]. These results were confirmed by MNDO, AM 1 and PM 3 calculations.

The introduction of substituent in the 5 position of triazines LXIII was performed by their converting into salts LXIV and the next radical nucleophilic carbamoylation followed by the elimination of dicyanomethylene [134—137]. Carbamoyl radical was generated using ammonium persulfate as a radical source.

The direct reaction of LXIII with nucleophiles would be impossible because of their instability in protic medium.

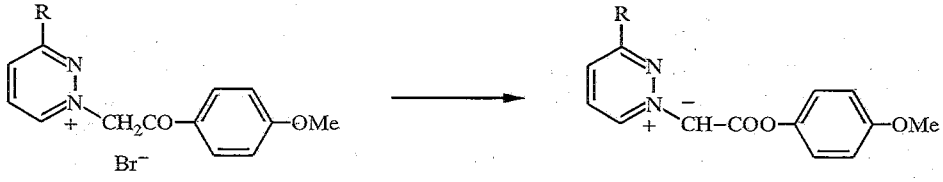


LXIII LXIV

R, R^1 = Me, Et, Ph

3. REACTIONS OF N-SUBSTITUTED AZAAROMATICS PROCEEDING ON THE NITROGEN ATOM AND ON SUBSTITUENTS AT THE LATTER

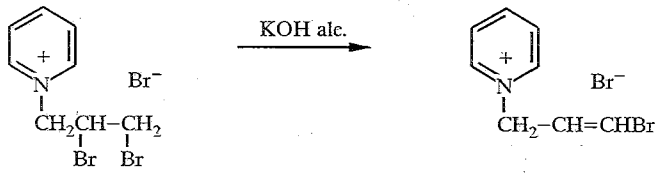
Among reactions of N-substituted azaaromatics proceeding on the nitrogen atom their dehydrohalogenation leading to ylides will be described, e. g. the dehydrobromination of pyridazinium bromides LXV in aqueous alkaline solution affording ylides LXVI [138].



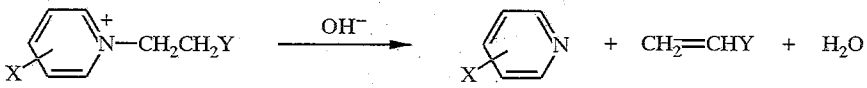
R = H, 4-Me-C_6H_4, 4-Cl-C_6H_4

LXV LXVI

Other example of dehydrobromination is the following reaction.



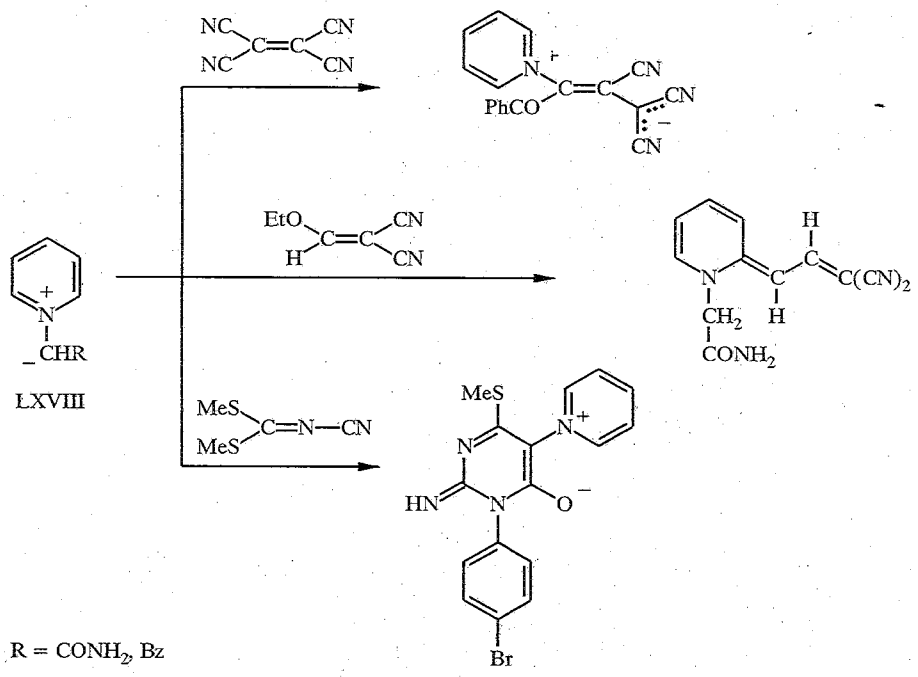
The hydroxide ion catalyzed elimination of pyridines from pyridinium cations LXVII was studied and its rate constants measured [140—143].



LXVII

X = H, 2-Me, 3-Me, 4-Me, 2-Cl; Y = CN, NO_2

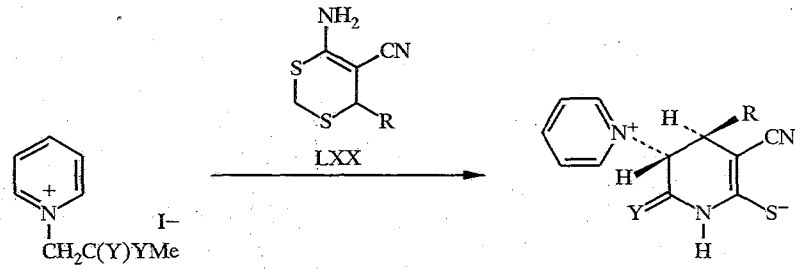
It was established that the following reactions of pyridinium ylide LXVIII are regio- and stereoselective [144—146].



LXVIII

R = $CONH_2$, Bz

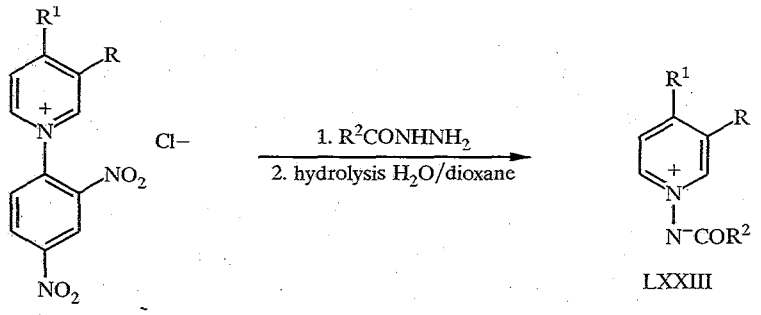
Pyridinium iodides LXIX react with dithiacyclohexenes LXX in the presence of 4-methylmorpholine to give pyridinethiolates LXXI. The reaction proceeds *via* corresponding pyridinium ylides [147].



LXIX LXXI

R = 4-FC_6H_4, 4-ClC_6H_4, 4-BrC_6H_4; Y = O, S

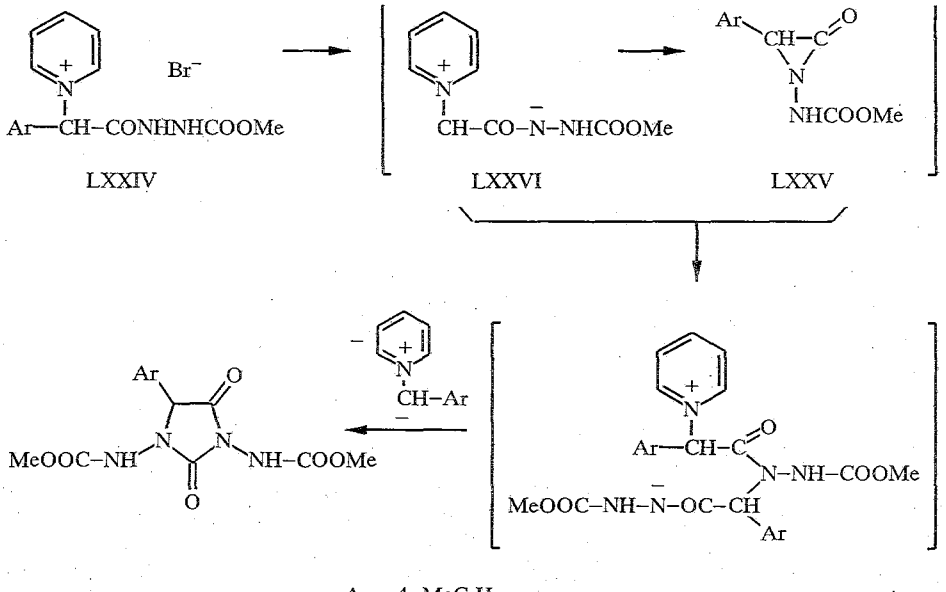
Treatment of pyridinium chloride LXXII with hydrazides and subsequent hydrolysis of the product affords pyridinium N-iminoylides LXXIII [148].



LXXII LXXIII

R, R^1 = H, Me; R^2 = Ph, 2-, 3-, 4-pyridyl

Hydrazido pyridinium salts LXXIV are starting materials for the easy synthesis of N,N'-diaminohydantoins; the following, rather unexpected mechanism of this process is proposed.

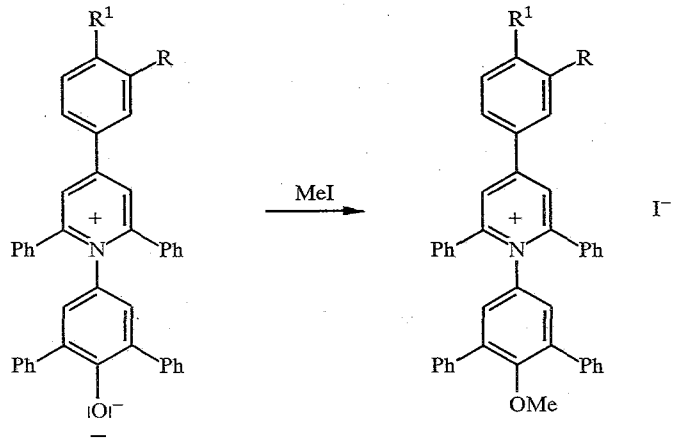


LXXIV LXXVI LXXV

Ar = 4-MeC_6H_4

The key step in the reaction is the nucleophilic ring opening of the aziridinone LXXV by the betaine LXXVI. Substituents of amino groups of the obtained hydantoin may be different [149, 150].

In the study of pyridinium phenoxide betaine dye LXXVIIa, the standard for determination of $E_T(30)$ values [151, 152] the methylation of betaine LXXVIIb by iodomethane in different solvents, leading to the salt LXXVIII was examined.

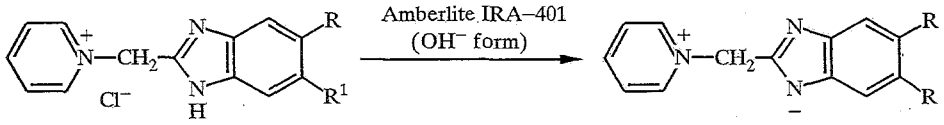


LXXVII LXXVIII

a $R=R^1=H$; b $R=H, R^1=Me$; c $R=CMe, R^1=H$; d $R=H, R^1=Cl$

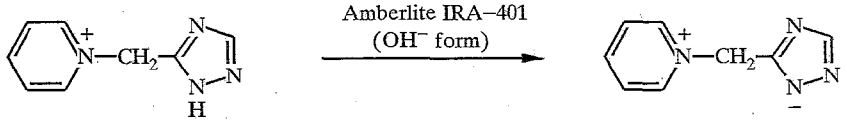
The reaction is of the pseudo-first-order; the effect of R, R^1 substituents can be described by a modified Hammett equation [153].

The salts LXXIXa—d and LXXX obtained by a quaternization reaction [154—157] were treated with anion-exchange Amberlite IRA-401 resin (OH^- form) [158] generating a necessary basic reaction medium to give betaines LXXXIa—d and LXXXII [159—165].



LXXIX LXXXIa–d

a $R=R^1=H$; b $R=Me, R^1=H$; c $R=H, R^1=HMe$; d $R=Me, R^1=HMe$

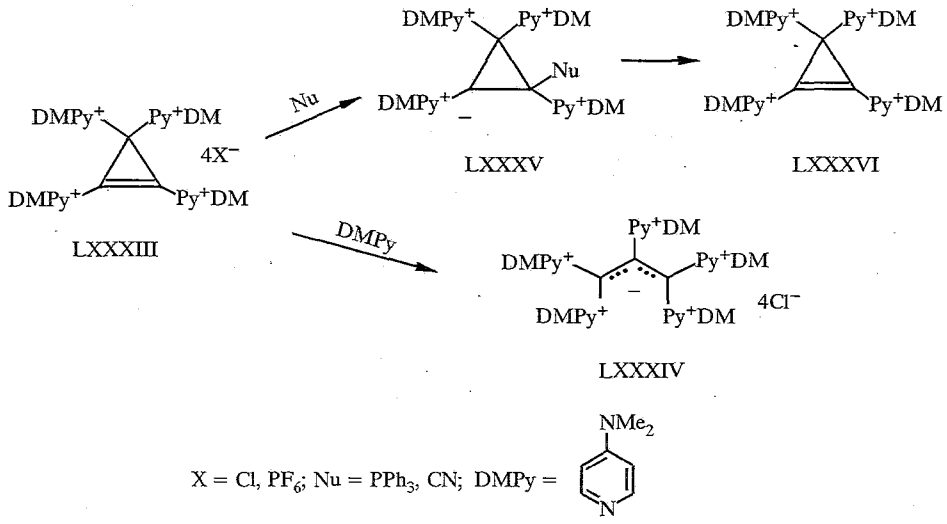


LXXX LXXXII

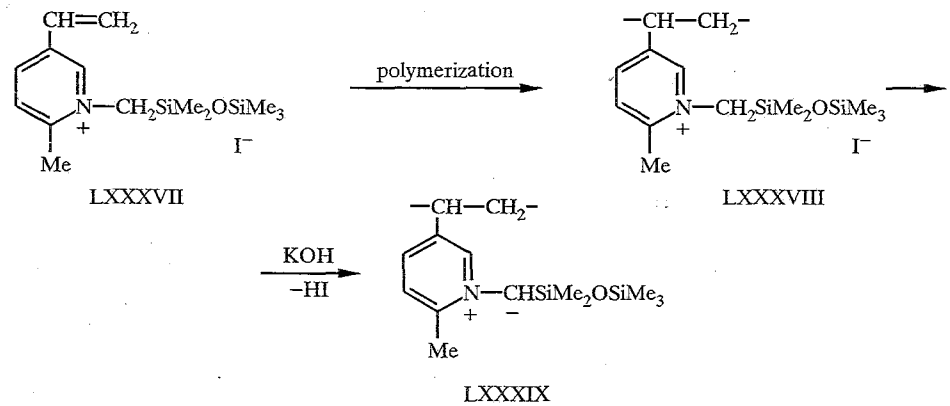
The formed betaines have high dipole moments; it was shown that their dipole moments calculated by AMI method are closer to experimental values than those calculated by MNDO method [159].

Cyclopropene derivative LXXXIII reacts with 4-(dimethylamino)pyridine (DMPy) to yield a very stable allylic anion LXXXIV. The reaction of LXXXIII

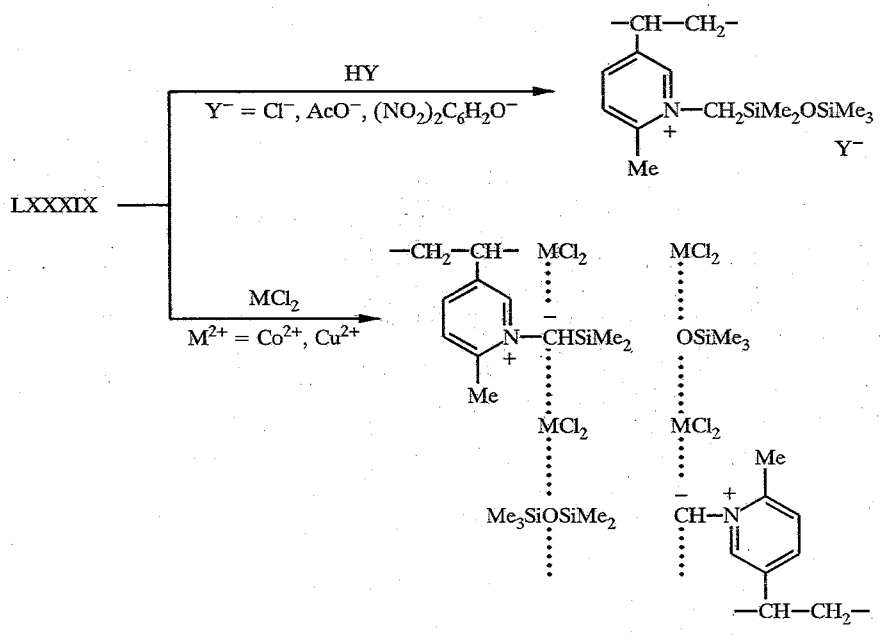
with other nucleophiles (e. g. PPh_3 or CN^-) proceeding *via* cyclopropyl anion intermediate LXXXV affords cyclopropene LXXXVI [166].



In the study of polymers bearing N-substituted azaaromatics, the polymerization of the monomer LXXXVII, leading to a water soluble polyelectrolyte LXXXVIII was performed. The treatment of LXXXVIII with alcoholic potassium hydroxide results in the formation of the stable ylide LXXXIX.



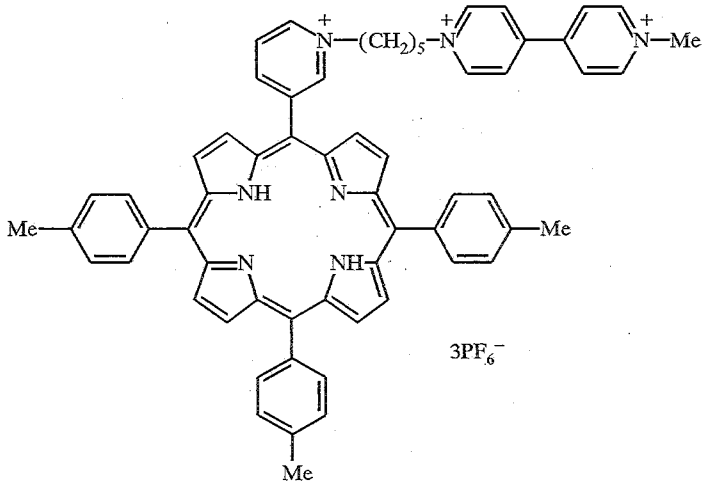
Due to the presence in LXXXIX of two electron donor centers — the ylide methine group and the siloxane oxygen atom it reacts with acids and with transition metal chlorides [41, 167].



4. REACTIONS OF PORPHYRINS BEARING N-SUBSTITUTED AZAAROMATIC MOIETIES

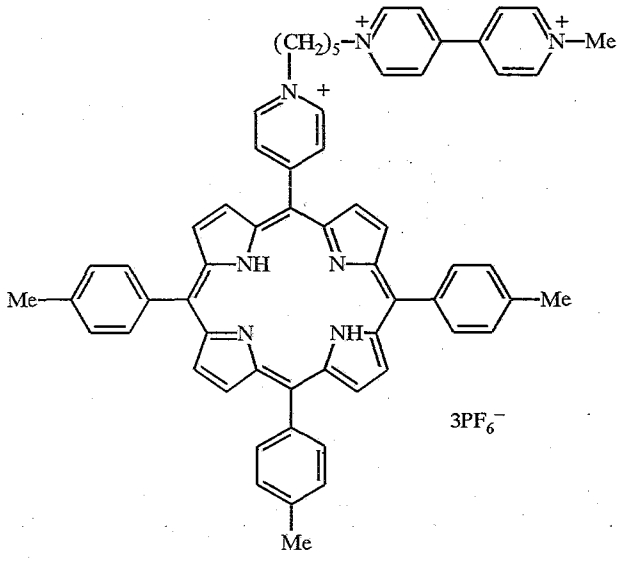
Porphyrins — free bases and metalloporphyrins bearing N-substituted azaaromatic moieties, especially viologens, are of interest as redox systems [168—176].

Examples of viologen linked porphyrins are LXX and LXXI, converted by treatment with $ZnCl_2$ into their zinc complexes.



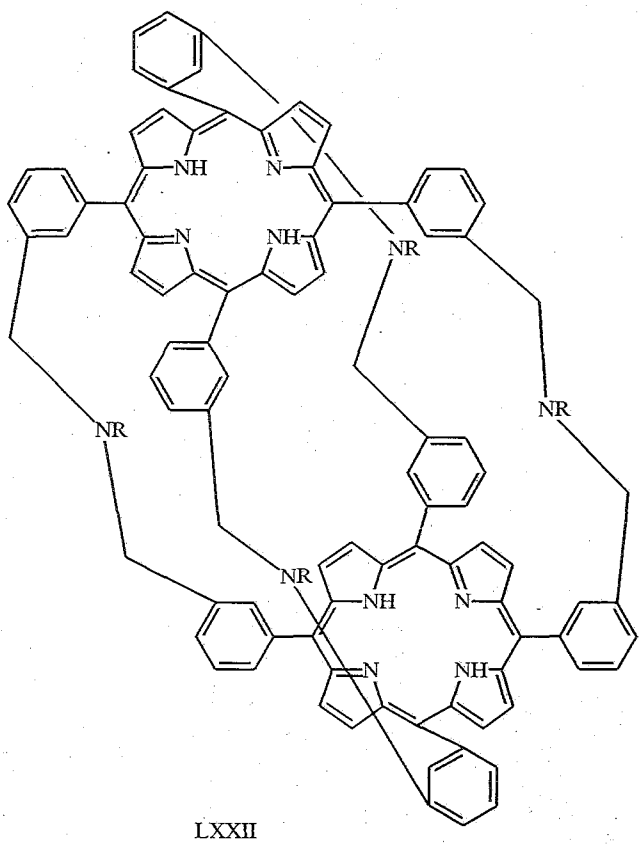
LXX

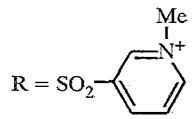
It is expected that in *meta*-compounds like LXX, the electron transfer from the porphyrin system to the viologen would be more rapid than in the case of *para*-compounds, like LXXI, due to the conformational effect [177].



LXXI

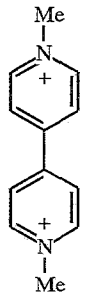
Water soluble, cofacial quadruply aza bridged bisporphyrins LXXII react with zinc diacetate to give corresponding bis-zinc complexes [178—180].



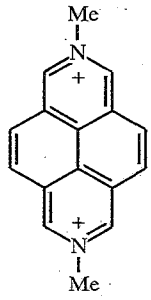


LXXII

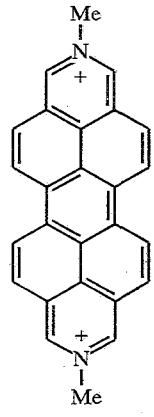
It should be mentioned here also ion pairs between N-substituted azaaromatics LXXIII—LXXV and negatively charged zinc *meso*-tetrakis(4-sulfonatophenyl)porphyrin formed spontaneously in water [181].



LXXIII



LXXIV



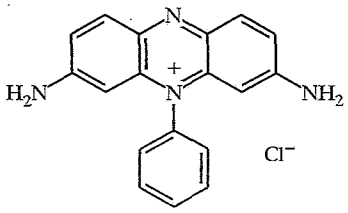
LXXV

The illumination of the ion pairs results in rapid electron transfer to form radical ion pairs decaying via charge recombination without dissociation [18, 181].

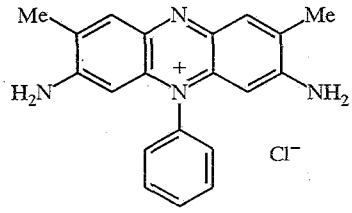
5. REACTIONS OF COMPLEXES OF N-SUBSTITUTED AZAAROMATICS

Numerous complexes of N-substituted azaaromatics are known [182—185] some examples will be described here.

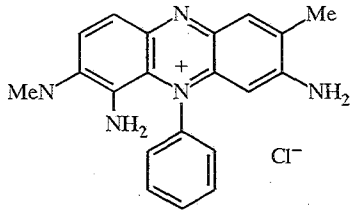
Cationic dyes LXXVI—LXXVIII interact strongly with anionic surfactants such as sodium octyl sulphate and sodium dodecyl sulphate even at very low concentrations (far below the cmc) [186]. The interaction products are protonated ion pairs induced by hydrophobic force.



LXXVI



LXXVII



LXXVIII

The photolysis of EDA complexes between methyl viologen (MV^{2+}) as acceptor and (*Z*)-stilbene or (*E*)-stilbene as donors has been performed in ethanolic and in micellar sodium dodecyl sulphate solutions, in the presence of oxygen.

It was shown that the stilbene loss quantum yields are influenced by MV^{2+} concentration and the acidity of media [187]. In the reaction mechanism the superoxide ion radical $O_2^{\cdot -}$ as an intermediate is proposed.

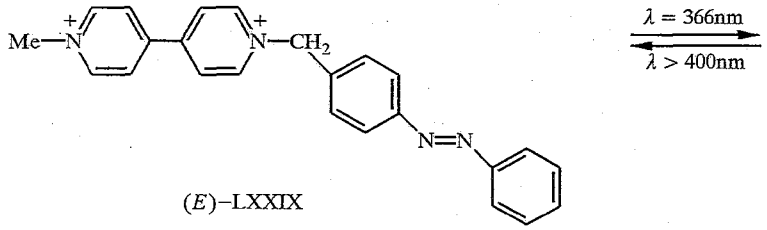
In the study of EDA complexes of viologens with xanthene dyes it was established that MV^{2+} forms with eosin 1 : 1 complex [Eo^{2-}—MV^{2+}], while in the case of Rose Bengal two complexes, i.e. [RB^{2-}—MV^{2+}] and [RB^{2-}—MV^{2+}——

RB^{2-}; MV^{2+}] are formed; in the latter one MV^{2+} unit is intercalated between two RB^{2-} units, and the second MV^{2+} unit is situated outside this sandwich structure.

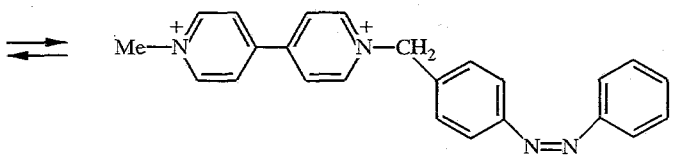
Similar Eo^{2-}—BV^{2+}—Eo^{2-}; BV^{2+} complex was obtained from benzylviologen and eosin [188—191].

The SiO_2 colloids separate ground-state complexes of bipyridinium-xanthene dyes by electrostatic interactions [191].

Azobenzobipyridinium diads (*E*)—LXXIX and (*Z*)—LXXIX form 1 : 1 complexes with eosin, which have different association constants, this fact being due to the photoinduced isomerization of LXXIX and change of its dipole moment [188, 192]. Compounds (*E*)—LXXIX and (*Z*)—LXXIX show photochromic properties; (*E*)—LXXIX irradiated with $\lambda = 366$ nm isomerizes into (*Z*)—LXXIX which at the irradiation with $\lambda > 400$ nm is again converted into (*E*)—LXXIX [188, 192].



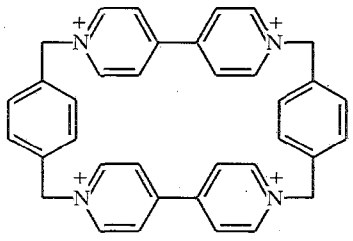
(*E*)–LXXIX



(*Z*)–LXXIX

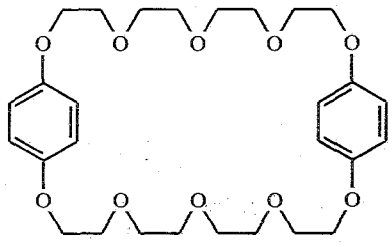
Having in view the rapid development of the host-guest chemistry, a special attention should be paid to host-guest complexes of N-substituted azaaromatics, where they play the role of a host or of a guest.

A great variety of systems where N-substituted azaaromatics serve as host molecules is known [193—195]; examples are complexes of cyclobis (paraquat-*p*-phenylene)tetracation LXXX incorporating isomeric dimethoxybenzenes [196, 197], tetrathiafulvalene [198] or amino acids with electron-rich aromatic moieties, e. g. tryptophan, tyrosine and phenylalanine [199].



LXXX

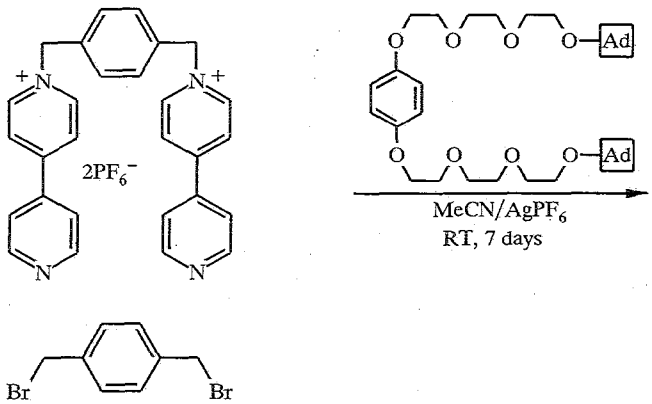
Among numerous systems where N-substituted azaaromatics are guests [200—202] the complex of paraquat and bis-*para*-phenylene [34]crown-10 LXXXI [203, 204] may be described.

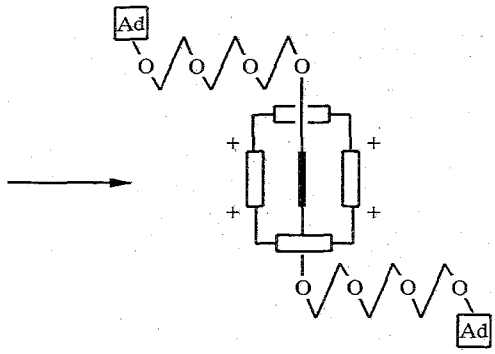


LXXXI

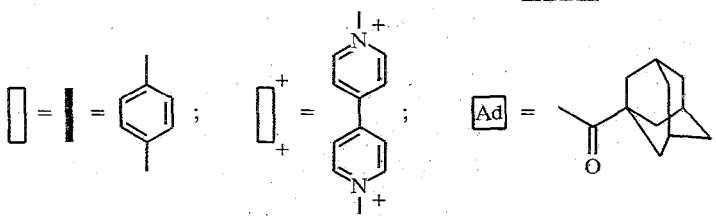
A subject of current interest are rotaxanes and catenanes where one unit is the N-substituted azaaromatic; a historical view on the development of these fascinating systems is given in ref. [205].

An example of a [2]rotaxane is LXXXII, obtained in the following way [206].



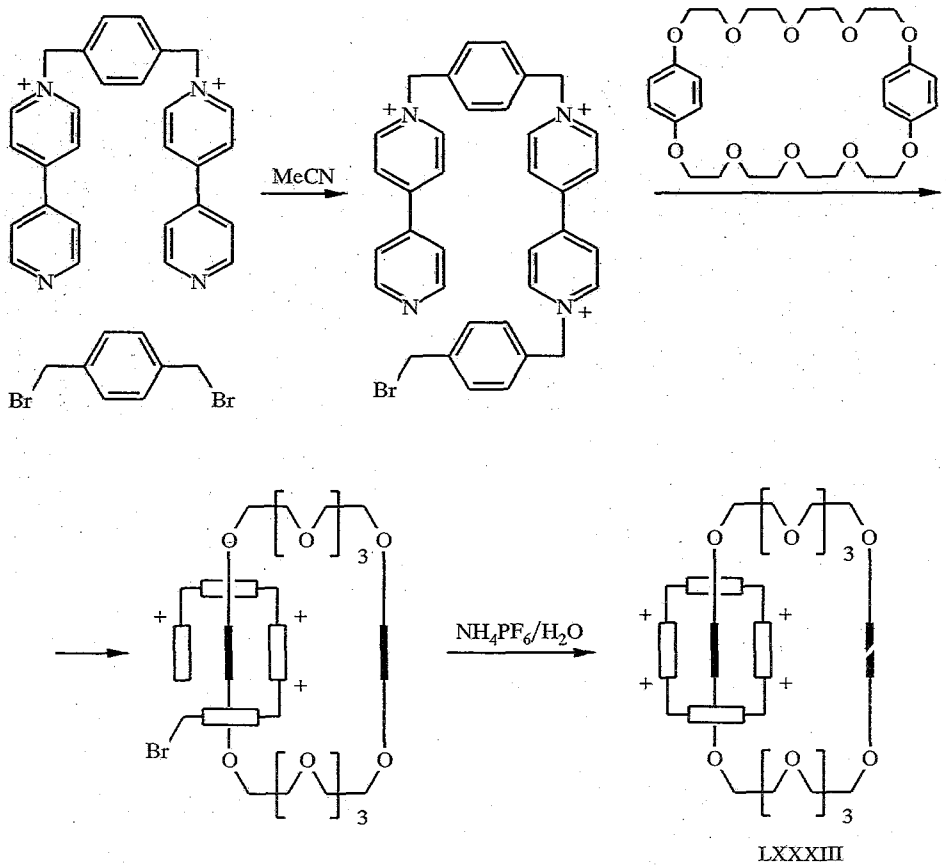


LXXXII



The tetracation LXXX may be compared to a bead, and the polyether chain to a thread; the bulky adamantyl groups serve as stoppers, preventing the bead from slipping off; among stoppers may be mentioned also triisopropylsilyl groups [206] or free-base metallated tetraarylporphyrins.

An example of a [2]catenane is LXXXIII synthesized in the following reaction [193, 199, 205, 207]:



LXXXIII

Numerous kinds of rotaxanes and catenanes are known [44, 45, 207—209]; their interesting property is the ability to behave like molecular shuttles or trains, i. e. in rotaxanes the bead moves to and fro along the thread, and in catenanes it makes a «course» around the polyether cycle; self-assemblies of this kind may be applied in molecular devices addressed photochemically [45, 205, 208, 210, 211].

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