CONDENSED HETEROAROMATIC SYSTEMS THAT INCLUDE π-SURPLUS AND π-DEFICIENT RINGS. AZAINDOLES (REVIEW)

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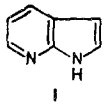
The results of our own research and the literature data on the principles of the construction of condensed heteroaromatic systems that include π-surplus and π-deficient rings and on the mutual effect of these rings are correlated.

It is known that the introduction of heteroatoms into an aromatic system substantially changes the distribution of the π-electron density, gives rise to certain inductive effects, and affects the polarizability of the substance, its capacity for solvation, and other physical and chemical properties. In 1958, A. Albert, in an examination of the methods for the classification of heterocycles, first advanced the concept of π-surplus character and π-deficient character of heteroaromatic systems [1]. If the heteroatom supplies two electrons to the aromatic system (as, for example, in pyrrole), the overall number of π electrons in the aromatic ensemble exceeds the number of atoms, and a certain excess amount of π-electron density develops on the carbon atoms. Albert proposed that such heteroaromatic systems be called π-surplus systems. When the heteroatom supplies only one electron for the creation of the aromatic ensemble (as, for example, in pyridine), the overall π-electron density on the ring carbon atoms is lower than on the heteroatom, and Albert called such heteroaromatic systems π-deficient systems.

In 1977-1979 in two exhaustive reviews [2, 3] published in Khimiya Geterotsiklicheskikh Soedinenii (Chemistry of Heterocyclic Compounds), A. F. Pozharskii made a detailed examination of the general concept of π-deficient character and π-surplus character of heteroaromatic compounds from the point of view of the creation of a unified scale for the quantitative characterization of effects and the possibility of the use for these purposes of calculated (quantum-chemical) or experimental (spectral, polarographic, kinetic, etc.) parameters. Unfortunately, the current level of theoretical organic chemistry still does not make it possible to work out sufficiently convincing and unambiguous criteria for the quantitative evaluation of these effects.

Somewhat earlier in the nineteen sixties, after having become familiar with the general concept of Albert, we set out to investigate the character of the mutual effect in condensed two-ring systems of π-surplus and π-deficient heterocycles using the isomeric pyrrolo[2,3-b]-, pyrrolo[3,2-b]-, pyrrolo[2,3-c]-, and pyrrolo[3,2-c]pyridines (I-IV), which were called 7-, 5-, 6-, and 4-azaindoles, as the subjects for such studies [4, 5].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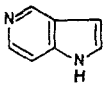
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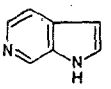
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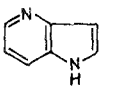
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In addition to the purely chemical interest involved, a study of this type has substantial value also from the point of view of the general principle of aza analogy as a method for the specific search for biologically active substances.

Our systematic studies of azaindoles over the last two decades have also revealed other aspects of this problem. The new type of cleavage of N–N bonds with simultaneous N-alkylation that we found in the course of construction of azaindole systems [6] was responsible

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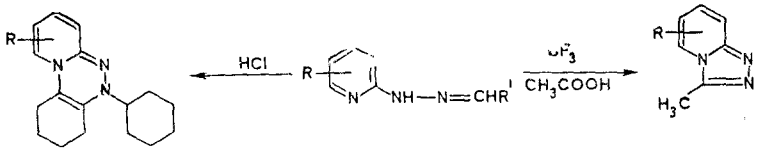
for the development of independent studies in this direction [7-17]. Azaindoles have been found to be convenient models for the in-depth study of the mechanisms of protolytic equilibria and the qualitative and quantitative characterization of the following transitions: free base + acid ⇄ molecular complex ⇄ ion pair ⇄ dissociated ions [18-21]. The photochemical transformations of azaindoles have led to the application of these compounds in silverless photography, including photoprocesses on polymeric materials, as well as in analytical chemistry. Nevertheless, the theme of this paper is a discussion of the problems involved in the synthesis and reactivities of the indicated compounds from the point of view of condensed two-ring systems that include π-surplus and π-deficient rings.

Synthesis of Condensed Heteroaromatic Systems That Include π-Surplus and π-Deficient Rings.

In the development of methods for the synthesis of isomeric azaindoles one must contend with the peculiarity of the electron density distribution in the condensed two-ring systems that contain π-surplus and π-deficient rings, as well as in the starting compounds for the preparation of systems of this type.

In the construction of azaindole structures on the basis of substituted pyridines with the formation of a pyrrole ring one must take into account the π-deficient character of the pyridine ring and the presence in it of a nitrogen heteroatom with increased electron density. Electrophilic substitution reactions, which lie at the foundation of most syntheses of indoles, have been found to proceed with greater difficulty on passing to azaindoles and require more severe conditions [4, 5, 22-39]. The possibility of competitive reactions at the electron-surplus nitrogen atom of the pyridine ring frequently becomes the principal direction of the process. This is due in a series of cases to errors in the establishment of the structure of the resulting substances, when compounds that have the pyrimidazole structure and the structures of other two-ring systems with a common nitrogen atom have been assumed to be azaindoles [40-44].

From this point of view, one of the chief methods for the synthesis of indole derivatives — the Fischer reaction (cyclization of arylhydrazones under acidic catalysis conditions) — gave very few positive results for the chemistry of azaindoles [4, 5, 22-34, 45-49]. This method, which is still being used today in the chemistry of azaindoles, usually leads to the desired products in low yields or generally gives negative results due to the relatively inert character of the carbon atoms of the pyridine ring with respect to electrophilic attack and the ease with which such attack occurs at the pyridine nitrogen atom. In particular, it has been shown that, instead of indolization processes, derivatives of pyrido[2,1-c]-as-triazine (V) and pyrido[2,1-c]-s-triazole (VI) are readily formed under the conditions of the Fischer reaction due to cyclization at the pyridine nitrogen atom [28, 50, 51].



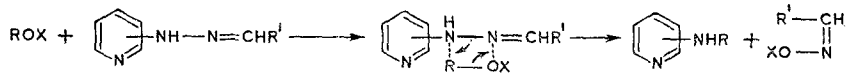
V

R' = cyclohexyl

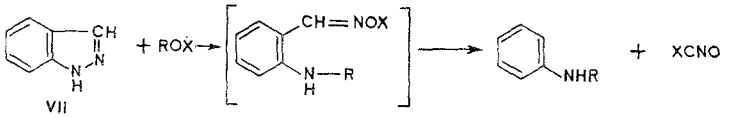
VI

The following modifications have expanded somewhat the possibilities of the Fischer reaction in the chemistry of azaindoles: "noncatalytic" thermal indolization [52-56] and indolization under conditions of high-temperature heterogeneous catalysis [57, 58]. It has also been shown that the Fischer reaction can be carried out in the presence of not only acidic catalysts but also alkaline catalysts (metal alkoxides) [7, 9]. It has been observed that in the latter case a process that competes with indolization is a new type of cleavage of the N—N bond to give monoalkylaminopyridines and syn-oximes of the carbonyl components of pyridylhydrazones [6, 10].

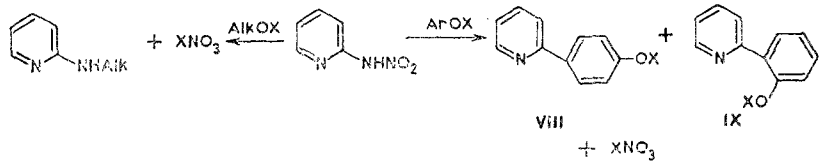
The reactions evidently are realized through four-membered transition states [10] via the scheme



The use of aromatic aldehydes, with which the "normal" course of the Fisher reaction is impossible, as the carbonyl components directs the process to favor the formation of N-monoalkylpyridines [10]. An increase in the polarity and polarizability of the N—N and R—O bonds by the introduction of substituents with various electromeric effects into the carbonyl and hydrazone parts of the molecule [10] and also by passing from Na alkoxides to K, Rb, and Cs alkoxides [16] has made it possible to control the course of this reaction and to extend it to other hetarylhydrazones of aromatic aldehydes [11, 59], diaryl(and aryl and hetaryl)hydrazines [15], N-nitroamino aromatic (heteroaromatic) compounds, and cyclic hydrazones of the indazole type (VII).

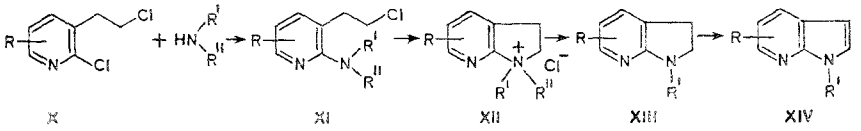


When the corresponding phenoxides are used in place of the alkali metal alkoxides, the reaction proceeds via a different (evidently radical) mechanism to give C-arylated pyridines VIII and IX.



In contrast to electrophilic substitution reactions, which are hindered in the π-deficient pyridine ring, the corresponding nucleophilic substitution reactions, especially in the α and γ positions of the pyridine molecule, are readily realized.

A general method for the synthesis of 5- and 7-azaindoles by the reaction of 3-(β-chloroethyl)pyridines that contain halogen atoms in the 4 or 2 position with ammonia and primary or secondary amines is also based on this peculiarity of pyridine derivatives [60-79].

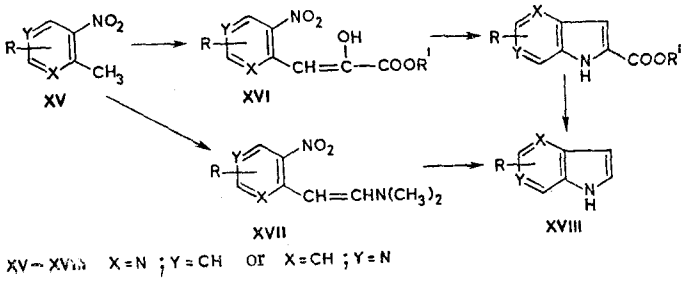


The reation begins with replacement of the halogen atom in the 2 position (or, respectively, the 4 position) of the pyridine ring by an amine residue, after which cyclization occurs, and the quaternary azaindolinium salt of the XII type that is formed when secondary amines are used splits out an alkyl halide to give a 7(or 5)-substituted azaindoline (XIII). This mechanism has been confirmed by means of the reaction of X with cyclic secondary amines [68], in which, due to breaking down of the process into its components, one can isolate products of each of the successive steps (XI and XII) and stop the synthesis at the stage involving the formation of a quaternary spiro derivative of the XII type. In the first step — replacement of the halogen atom in the pyridine ring by an amine residue — the lability of the halogen atom under attack, the reactivity of the attacking nucleophilic agent, which is associated with its basicity and steric factors, and the possibilities of competitive reactions (dehydrohalogenation or replacement of the chlorine atom by an amine residue in the β-chloroethyl chain) have a decisive effect. The effect of the reagent ratios, the reaction temperature and time, the polarity of the medium, and added catalytic salts has been studied quite thoroughly. The possibility of controlling the process by shifting it to favor the azaindoline derivative due to an increase in the polarity of the medium has been demonstrated [73]. It has been established that in experiments with unsymmetrical secondary amines one observes the characteristic (for the Hofmann cleavage of quaternary ammonium salts) ease of cleavage of the C—N bonds in an azaindolinium salt of the XII type, which increases in the order aryl < alkyl < benzyl [72], as compared with the order hexyl < ethyl < methyl for alkyl substituents [74]. The reaction has been extended to the synthesis of 5,7-diazaindole derivatives by using 4-chloro-5-(β-chloroethyl)pyrimidines [77].

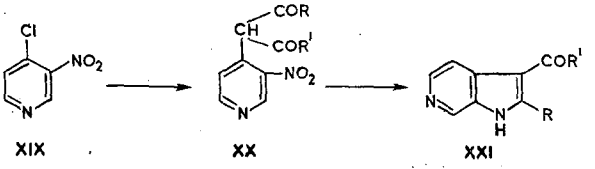
A study of the processes involved in the dehydrogenation of indolines, azaindolines, and diazaindolines to give completely aromatic compounds by means of quantitative methods of oxidative polarography on a platinum electrode and correlation of the $E_{1/2}$ values with the effects of substituents and data from preparative oxidation has made it possible to ascertain certain principles [80-88]. It has been shown that substances with $E_{1/2}$ less than 1.1 V are readily dehydrogenated by chloranil, substances with $E_{1/2}$ up to 1.2 V are readily dehydrogenated by dichlorodicyanoquinone, and substances with $E_{1/2}$ up to 1.4 V are readily dehydrogenated by activated manganese dioxide. Dehydrogenation is facilitated successively on passing from 5,7-diazaindolines to 5-azaindolines and 7-azaindolines and then to indoline compounds without an electron-acceptor pyridine ring. The transition from indolines or azaindolines to their N-metallated derivatives (i.e., to the corresponding anions) decreases the oxidation potential sharply, and dehydrogenation processes are realized readily by the action of air oxygen [87, 88].

Activation in the π-deficient ring of the α- and γ-methyl groups has been used [89-104] for the synthesis of the previously difficult-to-obtain 4- and 6-azaindoles, which cannot be obtained by the reaction of halopyridines that contain β-haloethyl groups with amines.

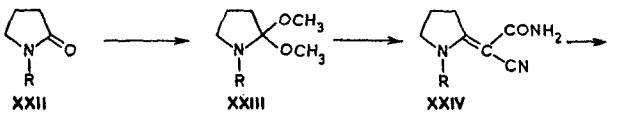
The condensation of o-nitropicolines XV with diethyl oxalate and subsequent Reissert reductive cyclization of XVI was initially used in these studies [89-92, 99-104]. However, the condensation of o-nitropicolines XV with dimethylformamide acetal rather than with diethyl oxalate and subsequent cyclization of nitro enamines XVII has proved to be more fruitful [93, 94, 97].

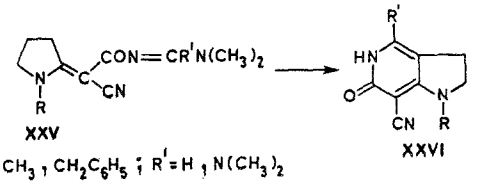


The use of o-nitropicolines substituted in the pyridine ring, as well as the corresponding o-nitropyridylacetones or o-nitropyridylacetic esters, has made it possible to obtain various derivatives of 4- and 6-azaindoles in high yields by this method [93-98]. Diverse substituted azaindoles have also been synthesized by the reaction of o-nitrohalopyridines XIX with β-dicarbonyl or β-cyanocarbonyl compounds (malonic, acetoacetic, benzoylacetic, and cyanoacetic esters) with subsequent reductive cyclization of the resulting XX or their transformation products [95, 98].

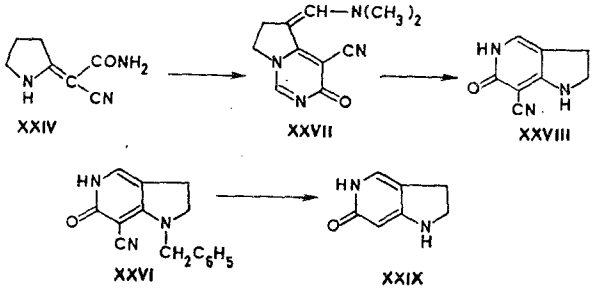


Syntheses based on 1-substituted 2-pyrrolidones (XXII) through acetal XXIII and the product of its condensation with cyanoacetamide XXIV and dimethylformamide acetal with subsequent cyclization of substituted acetylformamidine XXV have constituted a new direction in the synthesis of polysubstituted 5-azaindoles, but this time from the corresponding pyrrole derivatives with the construction of a pyridine ring [105-110].





The use of tetramethylurea acetal in place of dimethylformamide acetal has made it possible to obtain the previously unknown 1,4,6,7-tetrasubstituted 5-azaindolines and to pass to the synthesis of diverse compounds of this series that contain substituents in any positions of the 5-azaindole molecule. The formation of 6-oxo-7-cyano-2,3,5,6-tetrahydro-1H-pyrrolino[3,2-c]pyridine (XXVII) and its recyclization [110], as well as dibenzylation with the simultaneous decyanation of XXVI by the action of hydrogen bromide [107], have opened up possibilities for the preparation of 5-azaindole derivatives XXVIII and XXIX that are unsubstituted in the 1 position.



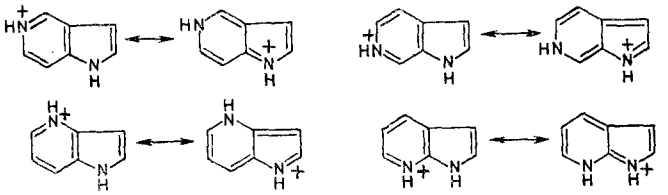
Peculiarities of the Chemical Properties of Condensed Heteroaromatic Systems That Include π-Surplus and π-Deficient Rings

Under our own initiative, in order to evaluate the mutual effect of π-surplus and π-deficient heterocycles in condensed systems we calculated the molecules of isomeric azaindoles, as well as indole, by means of the Hückel and Pariser—Parr—Pople methods [111-113]. The results constituted evidence for the relatively high π-electron charge on the C_3 atom for all of the azaindoles and for indole. The π-electron density distribution in the pyrrole part of the molecule on the C_1—C_2—C_3 atoms changed little on passing from one isomeric azaindole to another, i.e., rotation of the pyridine ring had only a slight effect on the π-electron structure of the five-membered ring. The π-electron charges in the six-membered ring rotated, as it were, when the position of the pyridine nitrogen atom changed without undergoing substantial distortion in the process. The maximum negative charge on the C_3 atom was also retained on passing to the cations of the isomeric azaindoles; the electron affinities increased in the order indole < 5-azaindole < 6-azaindole < 7-azaindole < 4-azaindole, whereas the ionization potentials increased in the order indole < 5-azaindole < 7-azaindole < 6-azaindole < 4-azaindole.

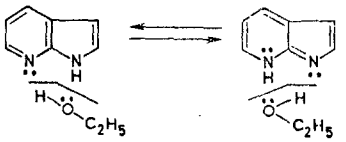
A study of the protonation of unsubstituted azaindoles has shown that the highest pK_a values are characteristic for 5-azaindole (8.26) and 6-azaindole (7.95), in which a large contribution of p-quinoid resonance forms is displayed [114]. The contribution of the o-quinoid resonance form is substantially smaller for 4-azaindole (6.94), whereas in the case of 7-azaindole [114], a strong additional inductive effect of the pyrrole nitrogen atom is displayed in addition to this, and the pK_a decreases to 4.59.

A study of the ionization constants of a large series of various azaindole derivatives by means of correlation methods has made it possible to correlate the principles of transmission of the effect of the substituents through the two-ring system and to calculate the transmission factors for these types of condensed π-surplus and π-deficient systems [115, 116].

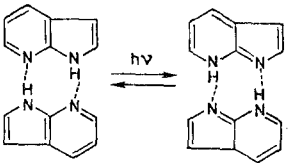
Processes involving the protonation of azaindoles in media with various dielectric constants have been investigated by means of PMR and IR spectroscopy [18-21], on the basis of which the contribution of quinoid structures with transfer of the positive charge to the pyrrole nitrogen atom has been evaluated.



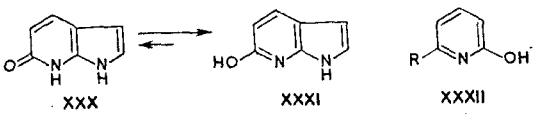
The determination in the PMR spectra of a dependence of the chemical shifts of the signals of the protons of the azaindole systems on the concentrations of acetic and trifluoroacetic acids in methylene chloride, acetonitrile, and deuteroacetone has made it possible to establish the relative position of the equilibria of reactions involving proton transfer from the donor to the acceptor through the formation of a hydrogen-bonded molecular complex of the base with the acid, which is followed by conversion to ion pairs and dissociated ions. In the case of 7-azaindole the 6-H proton, the strong-field shift of the signal of which on passing from the neutral molecule to the cation corresponds to the addition of a proton to the pyridine nitrogen atom with the formation of a conjugated amidinium ion, displays the greatest sensitivity to the effects of the medium and the formation of hydrogen bonds [18-21]. Biprotic phototautomerism has been observed for unprotonated 7-azaindole in ethanol by means of optical, absorption, and luminescence spectra [117-123].



In aprotic media similar proton transfer is realized on the basis of data from the fluorescence spectra, evidently through a hydrogen-bonded dimer [122].



Interesting results were obtained in a study of the lactam–lactim tautomerism of hydroxyazaindoles [124-130]. It is known [131-133] that α- or γ-hydroxy-N-heteroaromatic compounds are characterized by a sufficiently complete shift of the tautomeric equilibrium in solutions to favor the lactams and usually exist primarily in the oxo form. The presence of commensurable amounts of both forms, viz., the lactam (XXX) and lactim (XXXI) forms, is observed in the case of 6-hydroxy-7-azaindoles even in strongly polar solvents [125]. On passing to dioxane solutions the tautomeric equilibrium is shifted to favor the lactim forms so substantially that the carbonyl bands vanish completely in the IR spectra. A comprehensive investigation of this phenomenon — a quantitative evaluation of the effect of the polarity of the solvent on the tautomeric equilibrium constant, the character of the substituents attached to the pyrrole nitrogen atom and in other positions of the molecule, the reversibility of the process, the thermodynamic characteristics, etc. — has made it possible to establish that the inductive effect of the nitrogen atom of the pyrrole ring plays a significant role in this shift of the tautomeric equilibrium.



XXX XXXI XXXII

The overall pattern of the change in the tautomeric equilibrium is retained on passing to one-ring compounds XXXII; the effect of heterosubstituents in the 6 position of 2-hydroxypyridine XXXII increases in the order $R_2N < O < Cl$, in conformity with their —I effect [127].

Substantial weakening of the inductive effect of the nitrogen atom of the five-membered ring on passing from 6-hydroxy-7-azaindoles to 6-hydroxy(or 4-hydroxy)-5-azaindoles restores the characteristic (for α-hydroxy-N-heteroaromatic systems) shift of the tautomeric equilibrium virtually completely to favor the lactam forms [126, 130]. π-Electron interactions also have a definite effect on the position of lactam–lactim tautomerism in the investigated compounds [130]. The inductive effect of the nitrogen atom of the five-membered ring is retained on passing from 6-hydroxy-7-azaindole compounds of the XXXI type to the analogous 6-hydroxy-7-azaindoline derivatives, in which the inductive effect of the nitrogen atom of the five-membered ring is retained to a considerable degree, but the π-electron effects of the pyrrole ring are eliminated; this gives rise to a distinct shift of the lactam–lactim tautomeric equilibrium to favor the lactam form [124, 125].

The peculiarities of the lactam–lactim tautomerization of hydroxyazaindoles and their analogs [124-128] have generated considerable interest and have served as an impetus for the studies of other authors [134-141], including the study of the tautomeric equilibria in the gas phase [142-146], which have led to a significant re-examination of the general concepts of tautomerism [141, 144] and thereafter to a re-evaluation of the role of solvation effects for chemical reactions that take place in the liquid phase [147].

In the analysis of the reaction properties of isomeric azaindoles it should first of all be emphasized that information regarding the mechanisms of these reactions was almost completely absent up until recently, very few quantitative kinetic measurements had been made, and most of the conclusions had been based on the qualitative or semiquantitative characteristics of the processes. Nevertheless, one cannot fail to note some general principles in the chemistry of azaindole condensed π-surplus and π-deficient systems.

It follows from quantum-chemical calculations [111-113] that electrophilic substitution reactions in isomeric azaindoles are realized in the 3 position. The effect of the π-deficient pyridine fragment of the molecule is reflected in the certain difficulty encountered in these reactions as compared with the analogous processes in indoles [4, 5, 22]. Systematic experimental studies have shown that the isomeric 4-, 5-, 6-, and 7-azaindoles are capable of attack in the 3 position in nitration, halogenation, Friedel–Crafts acylation, cyanomethylation, and the Mannich and Vilsmeier reactions [89, 149-162]. The substituents attached to the pyrrole nitrogen atom and in the α positions relative to the pyridine nitrogen atom have a significant effect on the course of these reactions; the effect of the indicated substituents is determined not only by their direct electron-donor or electron-acceptor effect but also by the ability to hinder or facilitate protonation at the pyridine nitrogen atom, which, in turn, is responsible for the reaction of azaindole compounds with electrophiles in the free base or "conjugate acid" form.

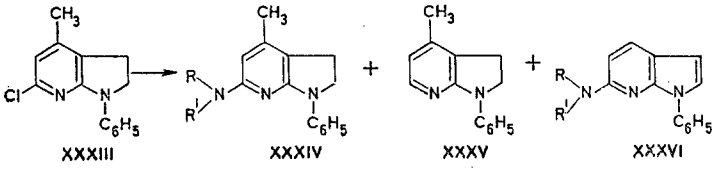
The initial purely qualitative studies did not reveal differences in the reactivities of the isomeric azaindoles in their reaction with electrophilic agents [89]. Finer kinetic studies of acid-catalyzed deuterium exchange in the 3 position of isomeric azaindoles by PMR spectroscopy [163] and nitration of the isomeric azaindoles in acidic media at low temperatures by means of polarographic analysis [164] have made it possible to reveal quantitative differences in the reactivities of 4-, 5-, 6-, and 7-azaindoles; it was shown that in conformity with the previously noted [114] greatest mutual effect of the pyridine and pyrrole nitrogen atoms in 5- and 7-azaindoles, which is associated, in particular, with the realization of p-quinoid resonance structures, the maximum rate of electrophilic substitution is observed for 5-azaindole, while 7-azaindole reacts somewhat more slowly. The 4- and 6-azaindoles undergo electrophilic substitution even more slowly, and the rates of nitration for these two isomers are very close [164]. The corresponding ionization potentials, which characterize the energies of the highest occupied orbitals of the C_3 atoms of the molecules under consideration, are the closest of all of the calculated indexes of the electronic structures of the isomeric azaindoles to the kinetic parameters [111-113].

In agreement with the results of quantum-chemical calculations [111], nucleophilic substitution processes in the isomeric azaindoles are realized in the α and γ positions relative to the pyridine nitrogen atom [4, 5, 22, 65, 124]; the effect of the pyrrole ring, which hinders nucleophilic substitution in the π-deficient fragment of the azaindole molecules, is substantially greater than the effect of the pyridine ring on electrophilic substitution reactions in the π-surplus fragment [124]. The isomeric azaindoles are not aminated under the conditions of the Chichibabin reaction [165, 166], which is so characteristic for compounds of the pyridine ring [167].

A methyl group in the γ position relative to the pyridine nitrogen atom of the azaindole molecule does not undergo condensation with aldehydes over a broad temperature range and is not oxidized by selenium dioxide [4, 5].

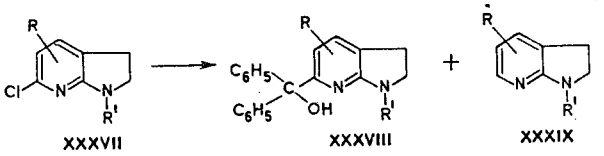
Replacement of the chlorine atoms with alkali metal alkoxides by alkoxy groups in the α position relative to the pyridine nitrogen atom in the isomeric azaindoles requires severe conditions [78, 108, 124]. Even more severe conditions are necessary for replacement of the indicated chlorine atoms by residues of primary or secondary amines [124, 168, 169], although autocatalytic effects for these reactions have been ascertained kinetically [170].

Reductive dehalogenation of the as yet unchanged XXXII by the developing N-substituted 1-phenyl-4-methyl-6-amino-7-azaindoline (XXXIV) with the simultaneous formation in equimolar amounts of 1-phenyl-4-methyl-7-azaindoline (XXXV) and N-substituted 1-phenyl-4-methyl-6-amino-7-azaindole (XXXVI) is observed along with the formation of the product of normal nucleophilic substitution (XXXIV) in the reaction of primary and secondary amines with 1-phenyl-4-methyl-6-chloro-7-azaindoline (XXXIII), which takes place under severe conditions [171, 172].

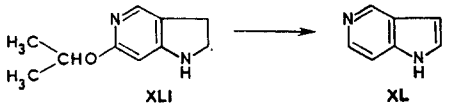


A similar reaction does not occur in the case of 5-azaindoline derivatives, which are characterized by a higher oxidation potential [168].

6-Unsubstituted 7-azaindolines (XXXIX) have also been obtained in the reaction of 6-chloro-7-azaindolines (XXXVII) with naphthyllithium under mild conditions (—40°C, tetrahydrofuran) and by subsequent treatment with benzophenone; substituted (7-aza-6-indolyl)-diphenylcarbinols (XXXVIII) are also formed in this reaction [173].



6-Unsubstituted 5-azaindole (XL) has been synthesized as a result of the reductive dealkoxylation of 6-isopropoxy-5-azaindoline (XLI) by dehydrogenation of the latter with a palladium catalyst [174].



As in the case of other heteroaromatic systems [130], the introduction of substituents in the α, α' positions relative to the pyridine nitrogen atom of the isomeric azaindoles and azaindolines changes their reactivities [130, 175-179].

In addition to the character of the condensation of the pyrrole and pyridine rings, the presence of substituents in the α position relative to the nitrogen atom of the pyridine ring also has a substantial effect on the ease of oxidation of the isomeric azaindolines to the corresponding azaisatins [180].

Thus, as one can see in the case of the isomeric azaindoles, this interaction between the indicated fragments of the molecule, which leads to certain hindrance to electrophilic substitution processes in the pyrrole ring and to a considerably greater degree suppresses processes involving nucleophilic substitution in the α and γ positions relative to the nitrogen atom in the pyridine part of the molecule, is characteristic for condensed hetero-

aromatic systems that include π-surplus and π-deficient rings. The character of the fusion of the π-surplus and π-deficient rings affects the manifestation of these peculiarities of the condensed systems, which is determined by the difference in the inductive interactions of the heteroatoms and the different contributions of the p- and o-quinoid resonance structures.

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