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CYCLIZATION OF N-ALKYLAZINIUM CATIONS WITH BIFUNCTIONAL NUCLEOPHILES. 15.* ISOMERIZATION OF THIAZOLO[4,5-b]QUINOXALINES IN THE PRESENCE OF ACIDS

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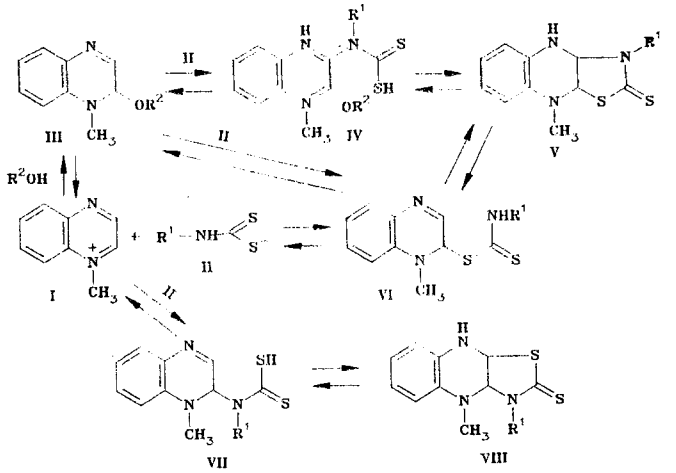
4-Alkyl-2-phenyl-3a,4,9,9a-tetrahydrothiazolo[4,5-b]quinoxalines undergo isomerization in chloroform in the presence of acids to the regioisomeric (with respect to them) 9-alkyl-substituted derivatives. Under the same conditions 2,4-dimethyl-3a,4-9,9a-tetrahydrothiazolo [4,5-b]quinoxaline undergoes isomerization to 4-methyl-1H-2,3,3a,4,9,9a-hexahydropyrrolo[2,3-b]quinoxaline-2-thione. It was demonstrated by means of deuterium labels that in both cases the isomerization proceeds through a step involving dissociation to a quinoxalinium cation and the corresponding thioamide.

Thanks to previous studies [2-13] that have been recently published, the cyclization of 2,3,5,6-tetrachloropyrazines and 2,3-dichloroquinoxalines with dinucleophiles can today be regarded as one of the general and quite attractive (because of its simplicity and accessibility) methods for the synthesis of 1,4-diazaaromatic systems that are condensed with imidazole [10, 11], thiazole [12], furan [13], pyran [13], thiazine [2], and other heterocycles.

The cyclization of quaternary N-alkylpyrazinium and quinoxalinium salts with dinucleophiles also leads to annelation of the pyrazine ring by various carbo- and heterocycles [14]; however, the products (formed as a result of diaddition) of cyclization with the tetrahydropyrazine ring differ substantially in their properties from their aromatic analogs. This is primarily manifested in the ability of hydrogenated polycyclic compounds to undergo dissociation in the presence of acids to give the starting substances; we have already noted that for derivatives of furo- [15] and pyrrolo[2,3-b]quinoxalines [16]. Another distinctive feature of the reactions of N-alkyl-1,4-diazinium salts with unsymmetrical dinucleophiles is the possibility of the formation of regioisomeric cycloadducts. Thus, depending on the condi-



*See [1] for Communication 14.

S. M. Kirov Ural Polytechnic Institute, Sverdlovsk 620002. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 3, pp. 396-403, March, 1985. Original article submitted March 22, 1984.

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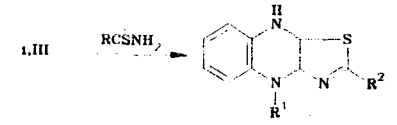
TABLE 1. Characteristics of Regioisomeric Thiazolo[4,5-b]quinoxalines IXb,c and Xa,b and Pyrrolo[2,3-b]quinoxaline (XI)

Compound	mp, °C (eec.)	Found, %				Empirical formula	Calc., %				Yield, %
		C	H	N	S		C	H	N	S	
IXb	90—92	68,8	5,8	14,2	10,8	$C_{17}H_{17}N_3S$	69,1	5,8	14,2	10,9	55
IXc	109—111	61,8	4,8	15,2	11,4	$C_{14}H_{13}N_3OS$	62,0	4,8	15,5	11,8	69
Xa	141—143	68,5	5,4	14,8	11,5	$C_{16}H_{15}N_3S$	68,3	5,4	14,9	11,4	70
Xb	144—145	69,2	5,8	14,3	11,1	$C_{17}H_{17}N_3S$	69,1	5,8	14,2	10,9	70
XI	174—176	60,5	6,0	18,7	14,6	$C_{11}H_{13}N_3S$	60,2	6,0	19,2	14,6	50

tions, either cycloadducts V or the regioisomeric (with respect to them) cyclization products VIII are formed in the reactions of the N-methylquinoxalinium cation (I) with N-alkyldithiocarbamates II [17].

We explain [17] the different specificities of the cyclizations in proton-donor solvents R^2OH (cyclization product V) and aprotic media (cyclization VIII) by the formation in an alcoholic medium of σ adducts III and their participation in cyclization via the III → IV → V pathway (or via the III → VI → V pathway), whereas in aprotic solvents the reaction proceeds via the I → VII → VIII pathway.

In contrast to dithiocarbamates II, thiazolo[4,5-b]quinoxalines with only one regio orientation (IXa-d) are formed in the reactions of thioamides both with cation I and with adduct III [18].

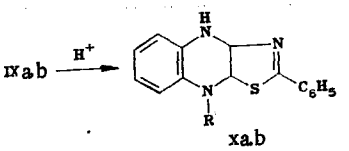


IXa-d

IX a,c,d $R^1=CH_3$; b $R^1=C_2H_5$; b $R^2=C_6H_5$; c R^2= furyl ; d $R^2=CH_3$

In the present research we established the fact of the isomerization of thizoloquinoxaline derivatives IXa-d under the influence of acids, and we also investigated the pathways of their isomerizational transformations as a function of the character of the R^2 substituent* and made an attempt to explain the other anomalous (at first glance) results of the reactions of quinoxalinium salts I with sulfur-containing dinucleophiles.

We established that in chloroform 4-alkyl-2-phenylthiazoloquinoxalines IXa,b undergo irreversible conversion to regioisomeric 9-alkyl-2-phenyltetrahydrothiazolo[4,5-b]quinoxalines (Xa,b) (Table 1).



Xab

X a $R=CH_3$; b $R=C_2H_5$

The structures of rearrangement products Xa,b were proved by means of the ^1H and ^{13}C NMR spectra, as well as by the mass spectra. Regioisomers IXa and Xa have identical m/z values of the molecular-ion peaks M^+ (281); however, their relative intensities and the character of the subsequent fragmentation differ (see the Experimental section).†

The presence of a $^3J_{CH}$ constant of splitting between the protons of the N-methyl group and the carbon-13 atom in the α position of the pyrazine ring makes it possible to differentiate the signals of the nodal $C_{(3a)}$ and $C_{(9a)}$ atoms in the ^{13}C NMR spectra of IXa and Xa, and the substantial difference in their chemical shifts makes it possible to determine the orientation of the thiazole ring. Thus the signal of the $C_{(3a)}$ carbon atom of Xa shows up

*We were unable to preparatively isolate or record in the PMR spectra a product of isomerization of thiazoloquinoxaline IXc, which has a furyl substituent in the 2 position; this is probably associated with the instability of furans in acidic media.

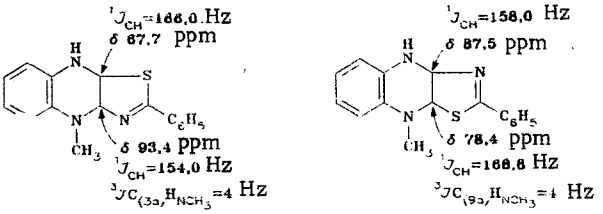
†We thank N. A. Klyuev (Moscow) for recording and interpreting the mass spectra.

TABLE 2. Spectral Characteristics of Regioisomeric Thiazolo[4,5-b]quinoxalines IXa-c and Pyrrolo[2,3-b]quinoxaline (XI)

Compound	Solvent	δ, ppm					J, Hz			ν_{NH}, cm^{-1}
		3a-H	9a-H	N—H	N—CH_3	aromatic protons	$^3J_{3a9a}$	J_{9a}	J_{3a4}	
IXa	d_6-DMSO	5,90 d	6,09 br d	6,20	3,10 s (3H)	6,4—6,8 m (4H) 7,3—7,8 m (5H)	7,0	—	—	3372
	$(CD_3)_2CO$	5,92 d	6,10 dd	5,45	3,20 s (3H)	6,5—6,7 m (4H) 7,4—7,8 m (5H)	7,0	1,0	—	
	CCl_4	5,65 d*	5,77 d*	3,60	3,20 s (3H)	6,5—6,7 m (4H) 7,3—7,7 m (5H)	—	—	—	
IXb	d_6-DMSO	6,00 s	6,00 s	6,19	3,56 q (2H) 1,31 t (3H)	6,4—6,8 m (4H) 7,2—7,9 m (5H)	—	—	—	3270
	$CDCl_3$	5,87 d	5,71 dd	3,81	3,65 q (2H) 1,40 t (3H)	6,4—6,9 m (4H) 7,2—7,9 m (5H)	6,0	1,0	—	
IXc	$(CD_3)_2CO$	5,85 d	6,02 dd	5,30	3,12 s (3H)	6,4—6,9 m (4H) 7,1—7,8 m (3H)	7,0	1,0	—	
Xa	d_6-DMSO	6,12 dd	6,29 d	—	2,79 s (3H)	6,5—6,8 m (4H) 7,2—7,8 m (5H)	8,0	—	1,5	3220
Xb	d_6-DMSO	6,06 br d	6,29 d	—	2,8—3,7 m (2H) 1,16 t (3H)	6,4—6,8 m (4H) 7,2—7,8 (5H)	8,0	—	—	3218
XI	d_6-DMSO	5,34 dd	4,21 dt ($^3J_{3,3a}$=6 Hz)	5,00	2,80 s (3H)	6,4—6,9 m (4H)	7,2	—	2	3368

*The assignment of these signals may be reversed. In $CDCl_3$ the signals of the 3a-H and 9a-H protons have identical shifts (see [18], as well as Fig. 1).

at 87.5 ppm as a doublet with $^1J_{CH}$ = 158 Hz, and a stronger-field signal of the $C_{(9a)}$ atom shows up at 78.4 ppm in the form of a doublet of quartets with $^1J_{CH}$ = 169 Hz and $^3J_{C(9a)HNCH_3}$ = 4 Hz. In the case of regioisomer IXa, however, the signals of the nodal carbon atoms, as already noted in [18], have opposite multiplicities. In both cases the nodal $C_{(9a)}$ atom, which is bonded directly to sulfur and nitrogen atoms, resonates at stronger field than the $C_{(3a)}$ carbon atom located between nitrogen atoms.



Appreciable differences are also observed in the ^1H NMR spectra of regioisomeric substances (Table 2). The 9a-H proton in the spectra of IX and X, which experiences the effect of the sulfur atom, resonates at weaker field than the 3a-H proton; however, the multiplicity of the 9a-H signal changes from a doublet in the case of 9-alkylthiazoloquinoxalines Xa,b to a doublet of doublets in the case of 4-alkyl-substituted isomers IXa,b due to coupling with the NH proton (Table 2). In the case of IXa the constant of spin-spin coupling between the NH protons and the adjacent CH group and is barely noticeable with respect to broadening of the signals in d_6-DMSO solution and is not visible at all in CCl_4 solution but shows up distinctly in d_6-acetone solution (Table 2). Let us also note the appreciable changes in the chemical shifts of the signals of the 3a-H and 9a-H protons of IXa on passing from d_6-DMSO and d_6-acetone to CCl_4 (∿0.3 ppm), the signal of the protons of the N-methyl group remains at δ 3.10-3.20 ppm, regardless of the nature of the solvent but undergoes a shift to strong field on passing to regioisomer Xa (Table 2).

These peculiarities of the ^1H NMR spectra make it possible to use this method to monitor the isomerization process. In particular, by means of the ^1H NMR spectra we showed that the isomerization of thiazoloquinoxalines IXa,b proceeds under the influence of not only acetic acid but also trifluoroacetic and hydrochloric acids (Fig. 1).

The isomerization of thiazoloquinoxaline IXd, which has a methyl group in the 2 position, proceeds in a different direction. The rearrangement results in the formation of 4-methylpyrrolo[2,3-b]quinoxaline-2-thione (XI), the structure of which was proved unambigu-

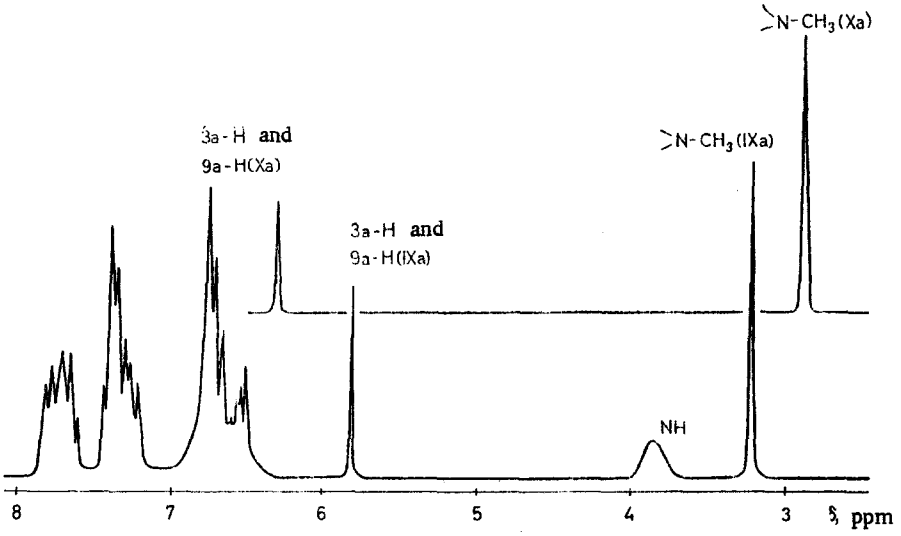
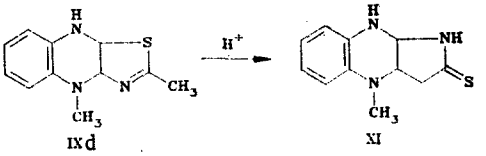
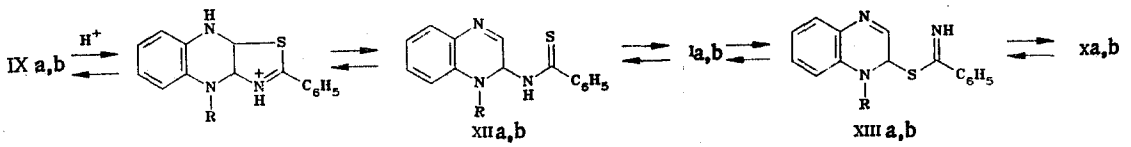


Fig. 1. Characteristic changes in the ^1H NMR spectra in the isomerization of 4-methylthiazolo[4,5-d]quinoxaline IXa to 9-methyl-substituted regioisomer Xa in $CDCl_3$ under the influence of trifluoroacetic acid.

ously by means of ^1H spectroscopy. The protons attached to the nodal carbon atoms (9a-H and 3a-H) have vicinal constant $^3J_{3a,9a} = 7.2$ Hz, which indicates their cis orientation; this is characteristic for annelation of five-membered heterocycles to quinoxalines [14]. In addition to this, the signal of the 3a-H proton is split by the protons of the CH_2 group, and the weaker-field signal of the 9a-H proton is split by the NH proton; this confirms the orientation in the fusion of the pyrrole ring with the pyrazine ring.



Inasmuch as in the isomerization of the thiazole ring to a pyrrole ring (IXd → XI), just as in the inversion of its orientation (IX → X), cleavage of two σ bonds (C—S and C—N) is inevitable, the question as to the sequency in which this occurs arises. If one proceeds from the analogies with respect to the reactions involving the diaddition of nucleophiles to quinoxalinium salts [19], one should assume that in the cyclic adducts and, in particular, in thiazolo[4,5-b]quinoxalines IXa-d the σ bond with the β-carbon atom of the pyrazine ring is weaker. The ease of splitting out of substituents from the β-carbon atom in N-alkyltetrahydroquinoxalines is due to the formation of a neutral azomethine bond, whereas dissociation at the α-carbon atom would lead a positively charged iminium salt. Thus the initiating step in the rearrangement is evidently the acid-catalyzed cleavage of the C—S bond in thiazoloquinoxalines IX, which leads to dihydroquinoxalines XII. This fact is confirmed experimentally by the fact that, after acetylation of the NH group of thiazoloquinoxaline IXd [18], it does not undergo isomerization, since cleavage of the C—S bond with a β-carbon atom is interlinked with the formation of a highly active N-acyliminium cation and requires large energy expenditures. The proposed isomerization scheme includes initial protonation of the $N_{(3)}$ atom in the double bond of the thiazole ring with subsequent cleavage of the C—S bond, i.e., through intermediate XII.



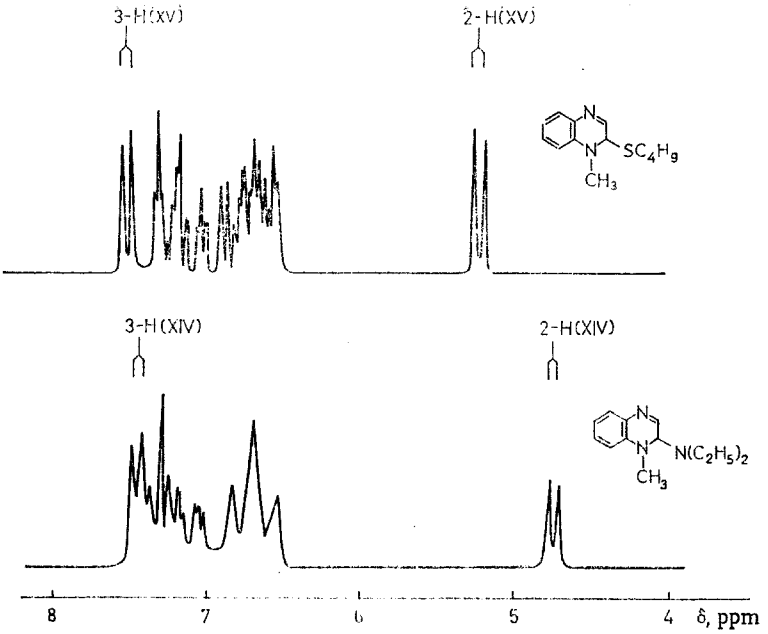
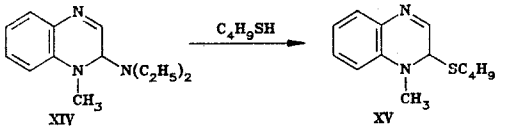


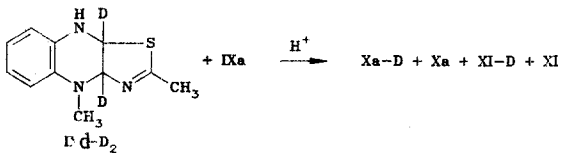
Fig. 2. Fragment of the ^{1}H NMR spectrum of 1-methyl-2-diethylamino-1,2-dihydroquinoxaline (XIV) in $CDCl_3$ (a) and the change (b) it undergoes when excess butyl mercaptan is added.

The participation of dihydro compounds XII in the rearrangement is even more likely if one takes into account the data in [19] regarding the ability of dihydroquinoxalines to substitute one residue in the α position for another by the action of nucleophiles. Proceeding from this, one might assume that the conversion of N-adduct XII to S-addition product XII lies at the foundation of the isomerization of IX to X. By modeling this step in the isomerization of thiazolo[4,5-b]quinoxalines we convinced ourselves that, in fact, the product of the addition of diethylamine to the quinoxalinium cation (N addition), viz., XIV, according to the data from the ^{1}H NMR spectra of solutions in deuterchloroform, is converted completely to S-adduct XV by the action of butyl mercaptan (Fig. 2).



XIV XV

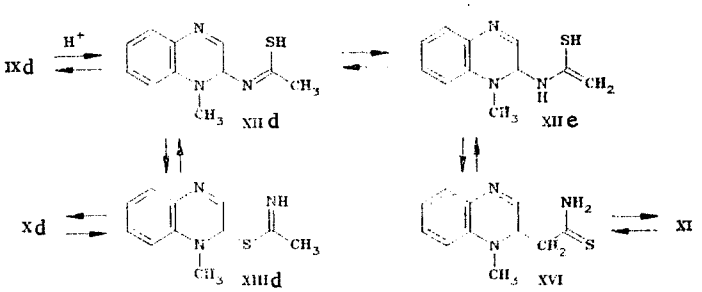
To arrive at an answer to the question as to whether this isomerization occurs intramolecularly (for example, via the scheme IX → XII → XIII → X) or includes a step involving dissociation to quinoxalinium cation I and a thioamide we carried out the following experiment. Thiazoloquinoxaline IXd-D_2 with deuterium atoms in the 3a and 9a positions was subjected to acidic isomerization in the presence of an equimolar amount of unlabeled thiazoloquinoxaline IXa (see the Experimental section). In the case of an intramolecular isomerization the reaction products would be thiazoloquinoxaline Xa with hydrogen atoms in the 3a and 9a positions and pyrroloquinoxaline XI-D_2 with deuterium labels. However, a mixture of isomerization products Xa and XI with an approximately equal distribution of the deuterium labels was observed.



IXd-D_2

Since deuterium exchange between tetrahydroquinoxalines IXa,d or Xa and XI is extremely unlikely,* the result of this experiment indicates that the thiazoloquinoxalines of both IXa and IXd undergo dissociation to give quinoxalinium cation I during isomerization.

*Deuterium exchange was not observed in the ^{1}H NMR spectra of IXa,d, Xa, and XI recorded in the presence of deuteroacetic acid, as well as deuteromethanol.

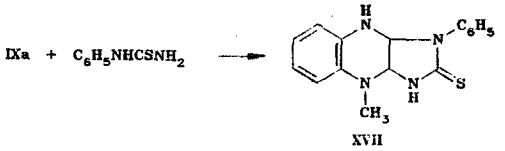


The mechanism of the isomerization of thiazoloquinoxaline IXd to pyrroloquinoxalinethione XI can be represented by a similar scheme with the difference being that σ-adduct XII exists in the tautomeric ketene-N,S-acetal XIIe form, as a consequence of which the primary pathway of its isomerization is the formation of C-adduct XVI with the subsequent formation of a pyrrole ring.

The possibility of the formation of thiazoloquinoxaline Xd from IXd in this reaction also exists, and the ^{1}H NMR spectra showed that it is actually formed at the initial instant of the reaction. Under the reaction (in an acidic medium) it is also capable of undergoing opening of the thiazole ring, and the reaction therefore ultimately leads to the more thermodynamically stable pyrroloquinoxaline XI, which can also be isolated preparatively in 50% yield.

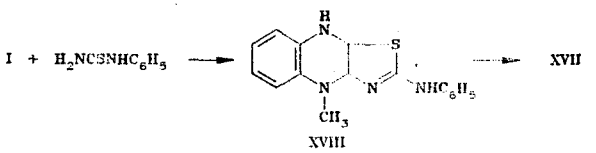
Thus the reason for the isomerizational transformations of thiazolo[4, 5b]quinoxalines IXa-d is evidently their different stabilities in an acidic medium.

The experiments show that hydrogenated thiazolo[4,5-b]quinoxalines can not only undergo isomerization but can also be converted to tetrahydroquinoxaline derivatives annelated with other five-membered heterocycles. Thus thiazoloquinoxaline IXa is converted smoothly to imidazo[4,5-b]quinoxaline-2-thione XVII when it is refluxed in ethanol with one equivalent of phenylthiourea.



This result makes it possible to understand the reason why the reactions of quinoxalinium salts with thioureas lead to annelation of the imidazole ring rather than the thiazole ring [20], i.e., thioureas display the properties of N,N'-rather than N,S-dinucleophiles, as observed in most cyclizations in which they participate [21].

It is apparent that in the cyclizations of salts I with thioureas under kinetic-control conditions it is also possible to observe the formation of thiazolo[4,5-b]quinoxalines XVIII, which, under the reaction conditions, are converted to the thermodynamically more stable imidazo[4,5-b]quinoxalines XVII.



Thus in the reactions of quinoxalinium salts with 1,3-dinucleophiles one should take into account the possibility of dissociation of the primary cycloadducts formed under kinetic-control conditions and their conversion to more thermodynamically stable substances; this may be accompanied not only by a change in the regio orientation but also in the very nature of the ring undergoing annelation.

EXPERIMENTAL

The ^{1}H NMR spectra of solutions of the compounds in deuterochloroform and d_6-DMSO were recorded with a Perkin-Elmer R-12B spectrometer (60 MHz) with tetramethylsilane (TMS) or hexamethyldisiloxane (HMDS) as the internal standards. The ^{13}C NMR spectra of solutions in d_6-DMSO were recorded with a Bruker WP-80 spectrometer (20.13 MHz) with TMS as the internal standard. The IR spectra of mineral oil suspensions were recorded with a UR-20 spectrometer.

Thiazolo[4,5-b]quinoxalines IXa,d were obtained by a previously described method, and their characteristics are presented in [18]. Compounds IXa,c (Tables 1 and 2) were similarly synthesized from the corresponding quinoxalinium salts and thioamides.

9-Methyl-2-phenyl-3a,4,9,9a-tetrahydrothiazolo[4,5-b]quinoxaline (Xa) (Tables 1 and 2). A 1-ml sample of acetic acid was added to a solution of 0.7 g (2.5 mmoles) of tetrahydrothiazoloquinoxaline IXa in 20 ml of chloroform, and the mixture was allowed to stand at room temperature for 1 h. The solvent was then evaporated *in vacuo*, and the precipitate was suspended in ethanol. The suspension was filtered, and the solid material was recrystallized from acetone to give light-yellow crystals of Xa with mp 141-143°C (dec.). The yield was 0.5 g (72%). ^{13}C NMR spectrum in d_6-DMSO, δ: 36.9 ($N-CH_3$); 78.3 [$C_{(9a)}$]; 87.5 [$C_{(9a)}$]; 112.7, 114.8, 119.3, 119.9, 127.9, 128.9, 131.7, 132.2. 134.9, 134.9, 135.8 (12 aromatic C); 163.7 ppm [$C_{(2)}$]. Mass spectrum (I ⩾ 20%), m/z; 50 (23), 51 (32), 76 (36), 77 (74), 77(74), 92 (22), 102 (22), 103 (51), 104 (41), 121 (44), 129 (34), 130 (30), 131 (100), 133 (25), 137 (37), 144 (98), 145 (98), 146 (70), 160 (85), 163 (94), 178 (40), 281 (M^+, 98).

Compound Xb (Tables 1 and 2) were similarly obtained.

4-Methyl-1H-2,3,3a,4,9,9a-hexahydropyrrolo[2,3-b]quinoxaline-2-thione (XI). A 1-ml sample of acetic acid was added to a solution of 1 g (4.6 mmoles) of thiazoloquinoxaline IXd in 30 ml of chloroform, after which the mixture was allowed to stand at room temperature for 1 h. The solvent was then evaporated *in vacuo*, and the precipitated XI was suspended in ethanol. The suspension was filtered, and the solid material was recrystallized from acetone to give 0.5 g (50%) of a product with mp 174-176°C (dec.).

9-Methyl-3-phenyl-2,3,3a,4,9,9a-hexahydro-1H-imidazo[4,5-b]quinoxaline-2-thione (XVII). A mixture of 1.5 g (5.3 mmoles) of thiazoloquinoxaline IXa and 0.81 g (5.3 mmoles) of phenylthiourea was refluxed in 25 ml of ethanol for 2 h. The next day, the precipitated imidazoquinoxaline-2-thione was removed by filtration and recrystallized from ethanol to give 1.2 g (75%) of a product that did not depress the melting point of a sample obtained from N-methylquinoxalinium iodide and phenylthiourea by the method on [20] and had an identical ^1H NMR spectrum.

Isomerization of a Mixture of 3a,9a-Dideutero-2,4-dimethyl-3a,4,9,9a-tetrahydrothiazolo[4,5-b]quinoxaline (IXd-D_2) and 4-Methyl-2-phenyl-3a,4,9,9a-tetrahydrothiazolo[4,5-b]-quinoxaline (IXa). A mixture of 32.2 mg (0.15 mmole) of deuterated IXd-D_2 and 40.2 mg (0.14 mmole) of tetrahydrothiazoloquinoxaline IXa was dissolved in 0.2 ml of $CDCl_3$, after which 20 μl of d_4-deuteroacetic acid was added. After 2.5 h, the ^1H NMR spectrum of the reaction mixture was recorded. Signals of undeuterated pyrroloquinoxaline-2-thione XI could be easily identified from the characteristic 9a-H doublet at δ 5.24 ppm (J = 7 Hz) and the doublet of triplets of the 3a-H proton at δ 4.12 ppm (J = 7 Hz and J = 6 Hz) (compare these values with data in Table 2). At the same time, the intensities of the signals of the nodal 3a-H and 9a-H protons of isomerization product X_a at 6.0-6.4 ppm are lower by a factor of two than expected with respect to the signal of the protons of the N-methyl group.

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