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CYCLOADDITION OF ANHYDRO BASES OF HETEROCYCLIC CATIONS.

1.* SYNTHESIS OF SPIROHETARENES IN THE REACTION OF s-TETRAZINES WITH THE ANHYDRO BASES OF ACRIDINIUM, XANTHYLIUM, AND THIOXANTHYLIUM SALTS

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UDC 547.815'818.6'835.-1'836.1'852.3'883

A new method of spiro-ring formation by the reaction of s-tetrazines with anhydro bases was described. The prototropic transitions were detected in the pyridazine fragments of the resulting spirocyclic compounds. The heterocycles of the anhydro bases are converted to cyclic ketones as a result of the breaking of the spiro junction in the acidic hydrolysis of the spirohetarenes. The regioselectivity of the cycloaddition was established.

The anhydro bases of the heterocyclic cations having the exocyclic double bond, which is not stabilized by acceptor substituents, are very reactive and extremely unstable organic compounds. They are sensitive to moisture and the oxygen of the air [1], are readily converted to dimers and polymers [2, 3], and are even carbonized [4]. The traditional chemistry of these compounds is associated with their participation in the synthesis of the cyanin dyes; moreover, the formation of the anhydro bases is often postulated in recyclization reactions [5]. We previously first found [6] the principle possibility of the participation of anhydro bases in the reactions of the cycloaddition to s-tetrazines, in which the s-tetrazines emerge in the role of reversed dienes, and the anhydro bases have the role of reversed dienophiles (the Carboni–Lindsey reaction [7]). The increased acceptor characteristics of the s-tetrazines [8] render them so powerful among the reversed dienes [9] that the requirement for the very laborious isolation of the monomeric anhydro bases becomes superfluous since the rates of their oxidation and dimerization are significantly less than the rates of cycloaddition; at the moment of formation, they are "intercepted" in a practically quantitative manner by the s-tetrazines. After the completion of investigations with the involvement of practically all classes of heterocyclic cations in the Carboni–Lindsey reaction [10], we are commencing the given series of the work in which exomethylene compounds of acridan, xanthene, and thioxanthene (A) are utilized as the anhydro bases. The choice of these compounds for the first communication is explained by the possibility of considering them as model fixed structures with the single "essentially double" [11] exocyclic bond determining the reactivity in the cycloaddition. Moreover, the presence of the very bulky phenylene fragments at such a double bond somewhat stabilizes their monomeric composition and, consequently, allows the experimentation with the isolated anhydro bases. Finally, the general features obtained by the study of the reaction with these model compounds will possibly be extended to the initial phase of the reactions of the anhydro bases of all the remaining heterocyclic cations.

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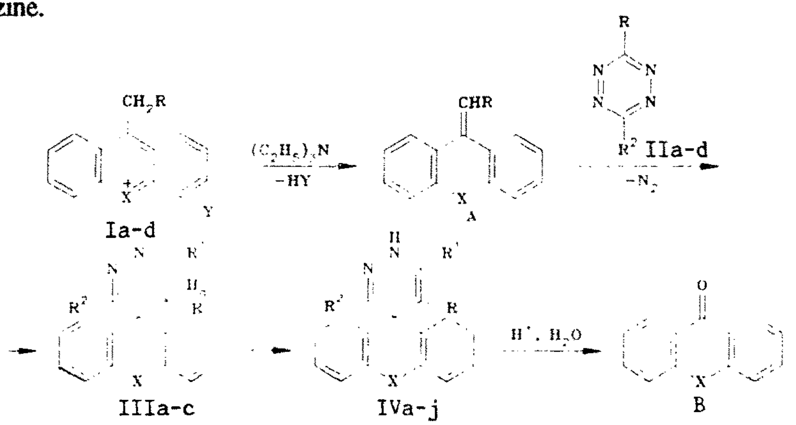
The methiodide of 9-methylacridine (Ia) reacts energetically with the symmetrically substituted s-tetrazines (IIa-c) on heating it or at room temperature in the presence of triethylamine. The reaction is readily monitored visually from the release of nitrogen gas and the disappearance of the red color characteristic of the s-tetrazines. The high-melting bright yellow or colorless substances (IVa-c) were obtained in almost quantitative yields (Table 1). The protons of the N-CH_3 groups in the compounds (IVa-c) absorb in the region of 3.35-3.55 ppm; this indicates the nonquaternized character of the nitrogen atom of the acridine, i.e., indicating the acridan structure of the compounds obtained (Table 2). The heteroallylic constant $^4J = 2$ Hz between the proton of the NH group and the proton of the CH of the pyridazine ring [12] disappears on the addition of deuteromethanol. Therefore, the data of the PMR spectra unambiguously determined the 1,4-dihydropyridazine structure (IV) of the spiro-linked pyridazine.



Compound*	R¹	R²	X	Y	Compound*	R¹	R²	X
Ia			NCH_3	I	IVa	α-C_5H_4N	α-C_5H_4N	NCH_3
Ib			NCH_3	I	IVb	C_6H_5	C_6H_5	NCH_3
Ic			O	ClO_4	IVc	C_6H_4Cl-*p*	C_6H_4Cl-*p*	NCH_3
Id			S	ClO_4	IVd	α-C_5H_4N	α-C_5H_4N	NCH_3
IIa	α-C_5H_4N	α-C_5H_4N			IVe	C_6H_5	C_6H_5	NCH_3
IIb	C_6H_5	C_6H_5			IVf	CH_3	C_6H_5	NCH_3
IIc	C_6H_4Cl-*p*	C_6H_4Cl-*p*			IVg	α-C_5H_4N	α-C_5H_4N	O
IId	CH_3	C_6H_5			IVh	C_6H_5	C_6H_5	O
IIIa	α-C_5H_4N	α-C_5H_4N	NCH_3		IVi	α-C_5H_4N	α-C_5H_4N	S
IIIb	C_6H_5	C_6H_5	NCH_3		IVj	CH_3	C_6H_5	O
IIIc	α-C_5H_4N	α-C_5H_4N	NCH_3					

In agreement with the traditional mechanisms of the cycloaddition to s-tetrazines [7], it was to be expected that the 4,5-dihydropyridazines (III), which also undergo isomerization to the more stable 1,4-dihydropyridazines (IV) and are characterized by homoaromatic character, should be formed directly after the cycloaddition and the subsequent release of nitrogen. In fact, the reactions of the tetrazines (IIa, b) with the benzene extracts of the anhydro bases (i.e., in the absence of triethylamine as the base) or with quaternary acridinium salts in DMF at low temperatures led to the isolation of the compounds (IIIa, b) which lack the NH group according to the IR and PMR spectral data, but have the two-proton singlet of the geminal protons at 5.30-5.35 ppm. The 4,5-dihydropyridazine structure of the compounds (III) is additionally confirmed for the compound (IIIc), where the SSCC of $^3J = 6$ Hz between the protons of the methyl group and the proton of the geminal position appears due to the removal of the degeneration of the geminal protons. Compound (IIIc) is formed very slowly at low temperatures in DMF, and the pyridazine (IVd) is obtained at once on heating it.

All the spirocyclic compounds (III) are unstable in the solutions and transform to the corresponding dihydropyridazines (IV). Under the usual conditions in chloroform or DMSO, this transition is accomplished virtually completely in 1 day, and not more than 2 min are required for this with the heating in o-dichlorobenzene. The isobestic point at 286 nm is clearly detected with the spectrophotometric monitoring of the tautomeric transition (IIIa) → (IVa) in ethanol at 50°C. The same process is observed in the thermogravimetric study of the pyridazines (IIIa, b). There is a single peak on the derivatogram of the compound (IIIb) with the maximum at 220°C, which is due to the isomerization to (IVb). The spiropyridazine (IIIa), containing the α-pyridyl substituents, undergoes such a transition at a lower temperature (200°C) whereby the initial substance of orange color is rapidly changed to the yellow compound (IVa), which melts at 220-225°C. The isomerization reaction of the pyridazines (III) to the compounds (IV) is facilitated in proton-donor solvents; it is catalyzed by acids and bases.

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*Ia, c, d, IIIa, b, IVa-c, f-j, R = H; Ib, IIIc, IVd, e, R = CH_3.

TABLE 1. Pyridazinospiro-4,9'-acridines (-xanthenes, -thioxanthenes)

Compound*	Empirical formula	mp, °C (ethanol)**	UV spectrum (ethanol), λ_{max}, nm (log ε)	Yield, %
IIIa	$C_{27}H_{21}N_5$	—	297 (4,46)	80
IIIb	$C_{29}H_{23}N_3$	—	253 (4,52)	73
IIIc	$C_{28}H_{23}N_5$	—	301 (4,41)	65
IVa	$C_{27}H_{21}N_5$	220...225	264 (4,38); 369 (3,57)	85
IVb	$C_{29}H_{23}N_3$	216...219	244 (4,47)	86
IVc	$C_{29}H_{21}Cl_2N_3$	269...272	260 (4,23)	94
IVd	$C_{28}H_{23}N_5$	218...220	254 (4,38)	91
IVe	$C_{30}H_{25}N_3$	226...228	241 (4,41); 329 (3,89)	94
IVf	$C_{24}H_{21}N_3$	263...266	237 (4,49); 288 (4,20); 326 (4,05)	91
IVg	$C_{26}H_{18}N_4O$	226...227	241 (4,48); 368 (3,61)	75
IVh	$C_{28}H_{20}N_2O$	209...210	238 (4,52); 354 (3,48)	85
IVi	$C_{26}H_{18}N_4S$	190...191	368 (3,64)	75
IVj	$C_{23}H_{18}N_2O$	205...207	346 (4,14)	68

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*Compounds (IIIa-c) undergo isomerization to the corresponding compounds (IV) on heating.

**Compounds (IVb) and (IVf) were crystallized from acetonitrile; (IVa) and (IVc) were crystallized from DMF, and (IVh) was crystallized from acetone.

TABLE 2. PMR Spectra of the Synthesized Compounds

Compound	Chemical shifts, δ, ppm (J, Hz)*				
	N—CH₃ (s.3H)	NH (br. ad d, 1H)	H_a, R, R^1	protons of α-pyridyls	benzene rings
IIIa	3,45	—	5,30 (s, 2H)	8,4...8,7	6,7...7,9
IIIb	3,45	—	5,35 (s, 2H)	—	6,7...7,8
IIIc	3,50	—	3,9 (., 1H), 0,70 (d, 3H); ($^3J=6$)	8,4...8,7	6,6...7,9
IVa	3,45	9,35	5,65 (d, 1H, $^4J=2,2$)	8,3...8,7	6,6...7,7
IVb	3,35	10,50	6,05 (d, 1H, $^4J=2,0$)	—	6,7...7,8
IVc	3,55	10,20	5,00 (d, 1H, $^4J=2,0$)	—	6,8...7,5
IVd	3,45	9,90	1,3 (s, 3H)	8,6...8,8	6,7...8,0
IVe	3,35	9,65	1,2 (s, 3H)	—	6,7...7,7
IVf	3,32	9,55	4,4 (d. q, 1H, $^4J=2,2$; $^4J=1,3$);	—	6,7...7,5
		($^4J=2,2$)	1,65 (d, 3H, $^4J=1,3$)		
IVg	—	9,50	5,70 (d, 1H, $^4J=2,0$)	8,15...8,80	6,9...8,0
IVh	—	9,08	4,93 (d, 1H, $^4J=2,1$)	—	6,7...7,7
IVi	—	9,35	5,73 (d. 1H, $^4J=2,0$)	8,15...8,60	6,7...7,7
IVj	—	9,7	4,5 (d. q, 1H, $^4J=1,9$, $^4J=1,3$);	—	6,7...7,5
		($^4J=1,9$)	1,85 (d, 3H, $^4J=1,3$)		

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spectra of (IVd-f) were recorded in DMSO-D_6.

The reactions of the s-tetrazines (II) with the anhydro bases of xanthylium and thioxanthylium salts (Ic, d) are characterized by certain features. For example, the anhydro base of 9-methylxanthylium (A; R = H, X = O) also reacts fairly smoothly with the more active tetrazine (IIa) and with the less reactive (IIb), whereas the anhydro base of 9-methylthioxanthylium (A; R = H, X = S) does not react with the tetrazine (IIb) either in the standard conditions or in the more drastic conditions. It is of special interest to note that the reaction between 3,6-di-(2-pyridyl)-s-tetrazine (IIa) and 9-methylxanthylium perchlorate (Ic) occurs on heating in alcohol without the addition of triethylamine. Even in the weakly basic ethanol, there is evidently an equilibrium between the salt form of (I) and the anhydro base (A), which is taken from the sphere of the reaction by the s-tetrazine. The equilibrium is strongly shifted in favor of the salt form since the initial compound remained quantitatively and qualitatively unchanged on the removal of the alcohol in the control experiment (without the tetrazine). Naturally, the more complete binding of the perchloric acid by the added triethylamine speeds up the reaction sharply.

In the spirocyclic compounds (IVg), (IVa), and (IVi), which represent a series with the monotonously changing electronegativity of the heteroatom involved in the formation of the ring (O → N → S), an anomalous antibatic character of the change in the chemical shifts of the sp^3-hybridized carbon of the spiro position is found: The higher the electronegativity of the hetero atom, the lower is the chemical shift (39.84, 43.11, and 45.08 ppm, correspondingly).

The cycloaddition reaction studied is characterized by high regioselectivity. The interaction of the unsymmetrically substituted tetrazine (IId) with the anhydro bases of 9-methylacridinium (Ia) and -xanthylium (Ic) results in the formation of high yields of the compounds (IVf) and (IVj). Their structure is shown by the presence of the doublet of protons of the methyl group and the quadruplet of the CH proton of the pyridazine ring with the SSCC $^4J = 1.3$ Hz in the PMR spectra. Therefore, the primary orientation of the methylene carbon of the exocyclic bond of the anhydro base occurs on the carbon atom of the s-tetrazine connected to the methyl group. Such a regioorientation may be explained by the fact that the exomethylene carbon atom of the anhydro base bears the greatest negative π-charge (the resonance consideration and the data of quantum-mechanical calculations), and the $C_{(3)}$ atom of the s-tetrazine (IId) connected to the methyl group has a higher positive charge (+0.141) by comparison with the $C_{(6)}$ atom connected to the phenyl (+0.081) (the calculations by the PPP method in the standard parametrization [13]; the parameters of the methyl group were taken from the work [14]). The indicated distribution of the positive charges in the s-tetrazine (IId) is confirmed by the attack with the single-centered nucleophile of hydroxide ions in the alkaline hydrolysis of the compound (IId), which any s-tetrazines are readily subjected to [15]. The treatment of the compound (IId) with an aqueous-alcoholic solution of sodium carbonate leads to the benzaldehyde acetylhydrazone (C), and not to the acetaldehyde benzoylhydrazone (D); this testifies to the attack by the hydroxide ion at the $C_{(3)}$ atom connected to the methyl group.

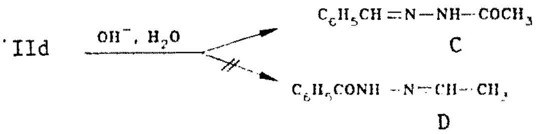
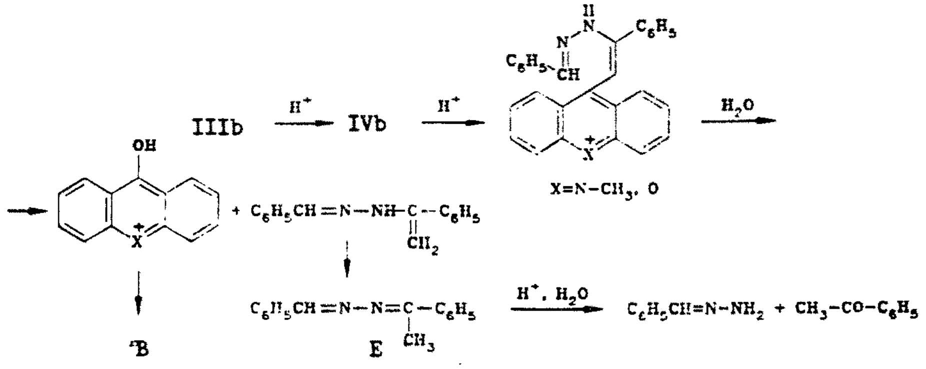


Photo- and thermochromic conversions characteristic of spirocyclic derivatives were noted for all the compounds (IV) [16]. In the light, the surface of their crystals is colored red; this color returns to the initial color in the dark or on heating the substance.

The spirocyclic compounds (III) and (IV) readily undergo the unexpected hydrolysis with the breaking of the spiro junction. For the compounds (IIIb) and (IVb, h), which have phenyl substituents in the dihydropyridazine ring, two ketones – acetophenone and the N-methylacridone (B) – could be found among the reaction products. The proposed scheme of the hydrolysis includes the isomerization of the compounds (III) to the pyridazines (IV) and the protonation of the dihydropyridazine ring accompanied by the breaking of one of the bonds of the spiro position and the formation of the N-methylacridinium (xanthylium) cation. This is followed by the hydrolytic cleavage of the C–C bond with the formation of N-methylacridone (xanthone) and the cleavage of the C=N bond of the mixed azine (E) with the formation of acetophenone.



EXPERIMENTAL

The IR spectra were taken using the UR-20 instrument and mineral oil. The electronic spectra were taken on the Specord UV-vis spectrophotometer. Thermogravimetric investigation was performed on the derivatograph of the type 3457 of the Paulik–Paulik–Erdey system.

The PMR spectra were recorded in deuterochloroform and DMSO-D_6 using the Perkin–Elmer R-12B (60 MHz) instrument. The PMR spectrum of compound (IVc) (due to its low solubility) and the ^{13}C NMR spectra of compounds (IVa, g, i) were recorded on the Bruker 80WP (80.13 MHz) spectrometer. The experiment on the double resonance for the compound (IVf) was accomplished on the same instrument. The internal standard in all the experiments was TMS.

The dihydro derivatives of the tetrazines (II) were obtained by the action of hydrazine hydrate on the thioanilide of picolinic acid (IIa) [17], the corresponding nitriles (IIb, c) [18], and the mixture of the hydrochlorides of acetamidine and benzamidine (IId) [19]. All the dihydrotetrazines were oxidized to the aromatic s-tetrazines (IIa-d) by sodium nitrite in acetic acid.

The acridinium (Ia, b), xanthylium (Ic), and thioxanthylium (Id) salts were synthesized according to [20], [21], and [22], correspondingly.

3,6-Di(2-pyridyl)-1,4-dihydro-10'-methylspiro[pyridazino-4,9'-acridan] (IVa). **A.** The suspension of 1 g (4.2 mmoles) of the s-tetrazine (IIa) and 1.5 g (4.5 mmoles) of the methiodide of 9-methylacridine (Ia) in 15 ml of ethanol is stirred with 0.7 ml of triethylamine until the discontinuance of the release of nitrogen bubbles and the disappearance of the red color of the tetrazine. The resulting pale yellow residue is filtered off and crystallized from DMF. The yield is 1.5 g (85%); the mp is 220-225°C.

The other **spirocyclic compounds (IV)** were obtained analogously (Tables 1 and 2). In a series of cases, it is possible to heat the suspension to boiling in order to accelerate the reaction; this does not exert influence on the yields of the reaction products.

B. The spirocyclic compound (IIIa) (0.5 g) is boiled in 3 ml of o-dichlorobenzene for 2 min. The resulting solution is cooled and treated with 5 ml of heptane. Compound (IVa) is precipitated quantitatively; it is identical in all its characteristics to the compound obtained according to method A. The same isomerization was observed for compounds (IIIb, c).

3,6-Di(2-pyridyl)-4,5-dihydro-5,10'-dimethylspiro[pyridazino-4,9'-acridan] (IIIc). The suspension of 1 g (4.2 mmoles) of the s-tetrazine (IIa) and 1.55 g (4.5 mmoles) of the methiodide of 9-ethylacridine (Ib) in 15 ml of DMF is stirred with 0.7 ml of triethylamine while cooling the mixture with ice water until the disappearance of the tetrazine from the reaction mass (monitoring by TLC). The resulting solution is diluted with water; the precipitated residue is filtered off, washed with ethanol, and dried in the air. The yield of compound (IIIc) is 1.2 g (65%).

The **4,5-dihydropyridazines (IIIa, b)** were synthesized analogously.

Acidic Hydrolysis of the Spirocyclic Compounds. Compound (IVb) (0.5 g) is boiled with 10 ml of 50% H_2SO_4 for 4 h. The solution which fluoresces strongly in UV light and has the characteristic smell of acetophenone is formed. The presence of acetophenone in the benzene extract of the reaction mass was shown by the method of GLC. The presence of N-methylacridone in the chloroform extract was shown by the method of TLC (Silufol, chloroform) with a "marker."

By analogy, the **ketones (B)** were obtained from compounds (IIIb) and (IVh).

Alkaline Hydrolysis of 3-Methyl-6-phenyl-s-tetrazine (IId). The s-tetrazine (IId) (0.86 g; 5 mmoles) is stirred with the solution of 2 g of sodium carbonate in 10 ml of water and 2 ml of ethanol until the discontinuance of the release of gas. The reaction mass, which smells strongly of benzaldehyde, is neutralized with acetic acid and concentrated to dryness in the air at room temperature. The solid substance is treated with water; the undissolved residue is filtered off and crystallized from the mixture of water and ethanol. The yield of 0.2 g (25%) of the compound (C) is obtained; it is identified as benzaldehyde acethydrazone (the direct synthesis is by the acetylation of benzaldehyde hydrazone).

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SYNTHESIS OF HETEROCYCLES ON THE BASIS OF PRODUCTS OF ADDITION OF POLYHALOALKANES TO UNSATURATED SYSTEMS.

2.* OBTAINING ISOXAZOLE DERIVATIVES BY 1,3-DIPOLAR CYCLOADDITION WITH THE PARTICIPATION OF POLYHALO-SUBSTITUTED ALKENECARBONITRILE OXIDES

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B. I. Ugrak, and M. M. Krayushkin

UDC 547.786.1.07'412.137'371.04:
543.422:541.621

Oximes of 3,3-dichloropropenal and 4,4,4-trifluoro-3-chloro-1-butenal were obtained on the basis of products of free-radical addition of CCl_4 *and* CF_3CCl_3 *to vinyl butyl ether. Generation of the corresponding nitrile oxides from these oximes and reaction of the nitrile oxides in situ with vinyl butyl ether, phenylacetylene, and propargyl alcohol via the scheme of 1,3-dipolar cycloaddition are proposed as a method for the synthesis of isoxazoles containing β,β-dichlorovinyl and β-trifluoromethyl-β-chlorovinyl substituents in the 3 position.*

The products of the addition of polyhaloalkanes to functionally substituted unsaturated systems are convenient starting compounds in the synthesis of various heterocycles. We have previously developed a new method for the synthesis of 1-aryl-5-chloropyrazoles by heterocyclization of arylhydrazones of 3,3-dichloro-2-propenal – an accessible unsaturated aldehyde obtained on the basis of products of addition of CCl_4 to vinyl ethers [1]. Later we subjected the production of addition of CCl_4 to methyl vinyl ketones – 3,5,5,5-tetrachloro-2-pentanone – to reactions that are characteristic for α-halo carbonyl compounds, which led to 2,2,2-trichloroethyl-substituted furans and thiazoles [2].

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*See [1] for Communication 1.

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