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STEREOCHEMICAL ASPECTS OF FORMATION OF SUBSTITUTED HYDROGENATED 3-(1-PYRIDINIO)-6-PYRIDINETHIOLATES AND SYNTHESIS OF 4,6-DIARYL-3-CYANO-2(1H)-PYRIDINETHIONES ON THEIR BASIS

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Regioselective and stereoselective methods were developed for the synthesis of 2-hydroxy-2,4-diaryl-3-(1-pyridinio)-5-cyano-3,4-trans-1,2,3,4-tetrahydropyridine-6-thiolates on the basis of the reactions of cyanothioacetamide with E-1-styrylpyridinium salts or aromatic aldehydes and 1-phenacylpyridinium bromide. The products exist in the half-chair conformation with the trans-diaxial arrangement of hydrogen atoms 3 and 4. The Michael adducts in the form of the anti conformers with the synclinal arrangement of the reaction centers act as intermediates. The obtained thiolates were converted with high yields into 4,6-diaryl-3-cyano-2(1H)-pyridinethiones.

As a result of the high polarization of the double bond by the pyridnium cation 1-styryl(vinyl)pyridinium salts enter into nucleophilic addition with amines, thiols, and CH acids [1] and also act as highly stereoselective dienophiles [2]. The ability of pyridine salts to undergo fairly ready transformation makes it possible to use them in the synthesis of the difficultly obtainable heterocycles 5-aza-3-oxatricyclo[4.2.1.$0^{2,6}$]non-7-enes,

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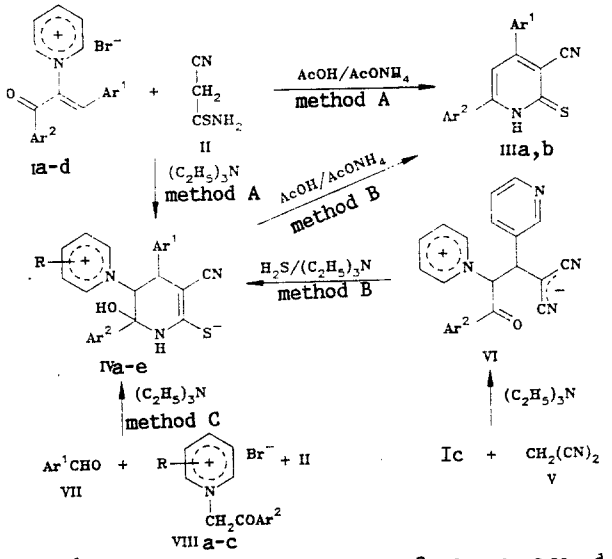
2-azabicyclo[3.3.0]octenes [3,4], pyrido[2,1-c]thiazines [5], and other compounds [6]. Until now, however, the reactivity of 1-styryl(vinyl)pyridinium salts has remained little investigated and has hardly been studied at all in reactions with the derivatives of cyanoacetic acid [7, 8]. In the present work we investigated the reactions of substituted 1-styrylpyridinium salts with cyanothioacetamide and established a relationship between the structure of the initial compounds, the intermediates, and the stereoselectivity of their transformation.

The reaction of the salts (Ia-d) with cyanothioacetamide (II) when boiled in acetic acid in the presence of ammonium acetate leads to 4,6-diaryl-3-cyano-2(1H)-pyridinethiones (IIIa, b) (method A). By changing the conditions of this reaction, we obtained the intermediates in the formation of the thiones (IIIa, b). Thus, the 2-hydroxy-2,4-diaryl-3-(1-pyridinio)-5-cyano-3,4-trans-1,2,3,4-tetrahydropyridine-6-thiolates (IVa-d) are formed in the reaction of the salts (Ia-d) with cyanothioacetamide (II) in ethanol in the presence of an equimolar amount of triethylamine (method A). Here the tetrahydropyridine-6-thiolates are converted into substituted 2(1H)-pyridinethiones (IIIa, b) when heated in acetic acid in the presence of ammonium acetate (method B). By changing the structure of the CH acid we were able to show that the pyridine-6-thiolates (IV) are formed through the corresponding Michael adducts. Thus, the substituted 3-(1-pyridinio)-1-propanide (VI) is obtained in the reaction of (Ic) with malononitrile (V) in ethanol. The subsequent reaction of the adduct (VI) with hydrogen sulfide leads to the tetrahydropyridine-6-thiolate (IVc) (method B).

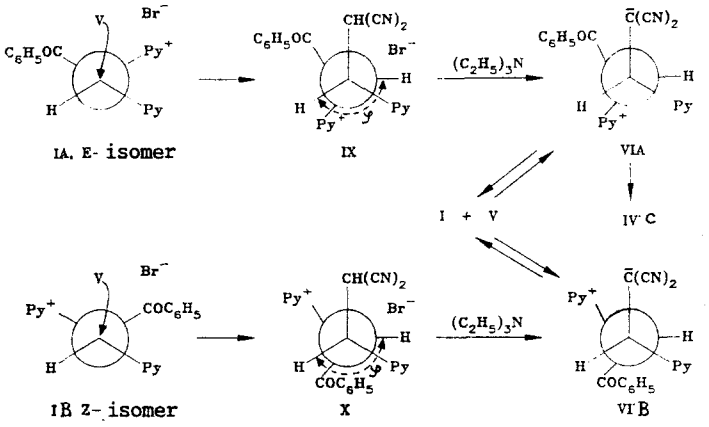
As a result of study of the above-mentioned reactions we developed a simpler method for the synthesis of substituted tetrahydropyridine-6-thiolates, which makes it possible to obtain them without previous isolation of the 1-styrylpyridinium salts. Thus, the three-component condensation of the aldehydes (VIIa-c), the pyridinium salts (VIIIa-c), and cyanothioacetamide (II) is highly stereoselective and leads to the formation of the tetrahydropyridine-6-thiolates (IVa-e) (method C).



Ia,d Ar^1=4-FC_6H_4, b Ar^1=4-BrC_6H_4, c Ar^1=3-C_5H_4N, a-c Ar^2=C_6H_5, d Ar^2=4-$BrCH_4$; III a Ar^1=4-FC_6H_4, b Ar^1=4-BrC_6H_4, a,b Ar^2=C_6H_5; IV a,d Ar^1=4-FC_6H_4, b,e Ar^1=4-BrC_6H_4, c Ar^1=3-C_5H_4N, a-c,e Ar^2=C_6H_5, d Ar^1=4-BrC_6H_4, a-d R=H, e R=2-CH_3; VII a Ar^1=4-FC_6H_4, b Ar^1=4-BrC_6H_4, c Ar^1=3-C_5H_4N; VIII a,c Ar^2=C_6H_5, b Ar^2=4-BrC_6H_4, a,b R=H, c R=2-CH_3

On the basis of a comparison of the data from physicochemical analysis of the 1-styrylpyridinium salts (I) [9] with data from analysis of (IV, VI) we were able to some degree to reveal a relationship between the stereoselectivity of the reactions leading to the formation of the substituted tetrahydropyridine-6-thiolates and the structure of the initial compounds. Compound (VI) is stabilized in the form of the pyridinium 1,4-ylide. In the IR spectrum of the adduct (VI) as a result of delocalization of the negative charge in the N...C...C...C...N^- fragment and of the asymmetry of the molecule there are two high-intensity absorption bands at 2102 and 2180 cm^{-1}. The first absorption band of (VI) is stronger and is shifted toward the low-frequency region by $\Delta\nu$ 173 cm^{-1} compared with the band of the malononitrile [10]. The absorption band of the C=O group lies in the region of 1690 cm^{-1}.

This indicates that delocalization of the negative charge in the $C_{(3)}$–C=O fragment of the molecules of (VI) must be excluded. In the PMR spectrum of the adduct (VI) the signals for the pyridinium protons are shifted downfield as a result of delocalization of the positive charge. The signals for the protons of the $C_{(2)}H$ and $C_{(3)}H$ groups appear in the form of two doublets in the regions of δ 4.13 and 5.37 ppm, respectively, with ^3J = 11.8 Hz. Using the dependence of the spin-spin coupling constant on the dihedral angle, we calculated the dihedral angle φ (161°) by means of the Karplus–Conroy equation. This presupposes the existence of two possible anti conformers (with respect to the arrangement of the hydrogen atoms). Their formation during the addition of malononitrile to the salt (Id) is undoubtedly determined by the steric structure of the investigated N-styrylpyridinium salt. Thus, the anti conformers (IX → VIA) with the synclinal arrangement of the reaction centers $C(CN)_2^-$ and COC_6H_5 are formed during the nucleophilic addition of malononitrile to the E isomer (IA) of compound (VI). The addition of malononitrile (V) to the Z isomer (IB) must lead to the anti conformers (X → VIB) with the anticlinal arrangement of the $C(CN)_2^-$ and COC_6H_5 groups. Here the conformational transitions (VIA) ⇆ (VIB) can only take place with the involvement of the Michael retroreaction. The experimental data indicate that the anti conformer (VIA) participates in the heterocyclization of (VI) to the tetrahydropyridine-6-thiolate (VIc). First, it was established earlier that the 1-styrylpyridinium salts obtained by the condensation of 1-aroylmethylenepyridinium salts with aromatic aldehydes [11, 12] exist in the form of the E isomer [9]. Consequently, the Z isomer (IB) does not participate in the reaction, and this rules out the possibility of the formation of the anti isomer (VIB).



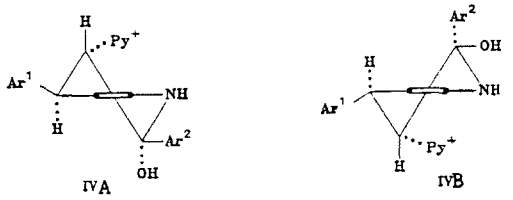
Second, it could be expected that the conformer (VIB) is formed with change in the direction of addition of malononitrile (V) to the isomers (IA, IB), but under the conditions of heterocyclization to the pyridine-6-thiolate (IVc) changes into the conformer (IVA), which then takes part in the investigated process. However, according to the data from PMR spectroscopy, no conformational transitions involving the reverse Michael reaction are observed when a solution of (VI) in DMSO-d_6 is heated from 20°C to 50°C. In the PMR spectrum of the adduct (VI) at 50°C there is a small degree of broadening in the signals of the protons in the $C_{(2)}H$–$C_{(3)}H$ fragment with slight narrowing of the components of the doublets and decrease in the spin-spin coupling constant to ^3J = 11.2 Hz. This leads to a decrease of the dihedral angle φ to 156°. In view of the fact that heterocyclization takes place at 15-20°C the possibility of the conformational transitions (VIA) ⇆ (VIB) can be excluded. Third, an essential condition in the cyclization of the adduct (VI) to the pyridine-6-thiolate (IVc) is the coplanar arrangement of the reaction centers COPh and $C(CN)_2^-$, since the reaction takes place stereoselectively with retention of the trans position of the protons in the $C_{(3)}H$–$C_{(4)}H$ fragment of the molecules of (IVc). Indeed the indicated stereochemistry favors stereoselective intramolecular heterocyclization during the transformation of one of the nitrile groups into the thioamide group in reaction of (VI) with hydrogen sulfide. The anticlinal arrangement of the COC_6H_5 and $C(CN)_2^-$ groups in the conformer (VIB) excludes the reaction path involving the formation of 3,4-trans-1,2,3,4-tetrahydropyridine-6-thiolates (IV).

According to the data from physicochemical analysis, compounds (IV) are stabilized in the form of betaines (Tables 1 and 2). The delocalization of the positive and negative charges in the Py^+ and N...C...C...S^- fragments of the molecules of (IV) leads to stabilization of the pyridinethiolates. As a result of delocalization of the negative charge in

TABLE 1. The Physicochemical Characteristics of 6-Hydroxy-4,6-diaryl-5-(1-pyridinio)-3-cyano-4,5-trans-1,4,5,6-tetrahydropyridine-2-thiolates (IVa-e)

Compound	Molecular formula	mp, °C	IR spectrum, cm^{-1}			Yield, %		
			OH, NH	C≡N	NH	A	B	C
IVa	$C_{23}H_{18}FN_3OS$	147...149	3205, 3398	2183	1630	87		82
IVb	$C_{23}H_{18}BrN_3OS$	141...143	3230, 3397	2179	1630	93		90
IVc	$C_{22}H_{18}N_4OS$	136...138	3192, 3415	2177	1632	67	72	89
IVd	$C_{23}H_{17}BrFN_3OS$	169...170	3250, 3385	2185	1630	83	94	94
IVe	$C_{24}H_{20}BrN_3OS$	140...142	3420	2183	1632			

the N...C...C...S^- fragment an increase in the intensity of the absorption band of the nitrile group with a simultaneous low-frequency shift to 2177-2185 cm^{-1} compared with the bands of the other substituted 2(1H)-pyridinethiones [13] is observed in the IR spectra of these compounds. In addition, the IR spectra of compounds (VI) contain absorption bands for the NH and OH groups (Table 1). The delocalization of the positive charge of the pyridinium leads to a downfield shift of the signals for the Py^+ protons compared with the signals of the recharged pyridines [14]. During comparison of the data from physicochemical analysis of the pyridine-6-thiolates (VI) with the data from x-ray crystallographic analysis and with the IR and PMR spectroscopic investigations of morpholinium and piperidinium pyridine-2-thiolates [15-18] it can be seen that as a result of the developed conjugation the N...C...C...S^- fragment is coplanar with the adjacent atoms. The $C_{(5)}$ and $C_{(6)}$ atoms lie on opposite sides of the ring. Thus, the pyridine-2-thiolates (IV), like cyclohexene [19] and the hydrogenated piperidinium pyridine-2-thiolates [17], exist in the half-chair conformation (IVA, B).



The signals of the $C_{(4)}H$ and $C_{(3)}H$ protons appear as two doublets in the regions of 4.48-4.66 and 5.18-5.23 ppm, respectively, with ^3J values of 11.5-11.9 Hz (Table 2). For the indicated values of the spin-spin coupling constants the torsion angles φ calculated by means of the Karplus–Conroy equation [14] lie in the range of 158-162° (Table 2). This indicates that the 3-H and 4-H hydrogen atoms are in the trans-pseudodiaxial arrangement. It should be noted that the indicated arrangement of the substituents is indeed the most favorable. The signals for the protons of the OH and NH groups appear as a broad signal, which in the PMR spectra (Table 2) disappears with the addition of deuterated water to a solution of compounds (IV) in DMSO-d_6.

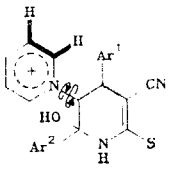
Determination of the steric arrangement of the substituents Ar^2 and OH is not possible at the given stage of the investigations, and two conformations (IVA) and (IVB) can therefore be proposed for compounds (IVa-e). In practice only one of them is realized. On the basis of the PMR spectra the pyridinio substituent in the molecules of (IVa-d) undergoes vibrational movements about the axis of the N–$C_{(3)}$ bond, and this leads to broadening of the signals for the α- and β-protons of the pyridinium (Table 2). The signals of the γ-protons appear in the form of a triplet in the region of 8.32-8.34 ppm with ^3J = 7.7 Hz. The rotation of the substituent is excluded as a result of the engagement of the Py^+ substituent with the adjacent substituents. The construction of models of compounds (IV) indicates that the conformations related to the arrangement of the substituents Ar^1 and Py^+ are interrelated, and the angle of rotation of the pyridinium about the $C_{(3)}$–N bond to one or the other side cannot exceed 80-90°. The vibrational rotations and the equatorial position of the pyridinium give rise to screening of at least half of the α- and β-protons (shown in bold type), and this leads to an upfield shift of their signals. The other part of the α- and β-protons of the pyridinium (normal type) is under the descreening influence of the pyridinethiolate system, and their chemical shifts are moved downfield (Table 2). The

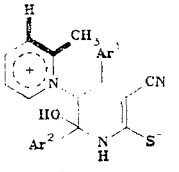
TABLE 2. The Parameters of the PMR Spectra of Thiolates (IVa-e)

Compound	PMR spectrum, δ, ppm (DMSO-d_6)								SSCC	Torsion angle
	protons of pyridinium cation*				Ar, m	NH, OH. br. s	$C_{(3)}H$, d	$C_{(4)}H$, d	$^3J_{4,5'}$ Hz	ψ°
	1H, br.s 5-H	2H, br.s 3-H, 6-H	1H,t; 4-H (^3J=7,7 Hz)	1H, br.s 2-H						
IVa	7,52	8,03	8,32	9,51	6,97...7,30	5,94	5,19	4,66	11,7	160
IVb	7,52	8,02	8,32	9,52	7,17...7,36	5,92	5,19	4,65	11,7	160
IVc	7,58	8,02	8,34	9,48	7,14...7,34	5,90	5,18	4,48	11,9	162
IVd	7,58	8,04	8,34	9,47	6,95...7,38	6,14	5,23	4,64	11,7	160
IVe**					7,20...7,37	5,59	5,19	4,63	11,5	158

*Signals for the protons of 3-C_5H_4N, δ: 7.21 (1H, q, 5-H); 7.64 (1H, d, 4-H); 8.29 ppm (2H, m, 2-H, 6-H).
**For IVe δ: 7.44 (1H, d, 3-H, ^3J = 7.9 Hz); 7.98 (1H, t, 5-H); 8.27 (1H, t, 4-H), 9.65 ppm (1H, d, 6-H, ^3J = 8.5 Hz). Signals for the protons of CH_3, δ: 1.80 ppm (3H, s).

accuracy of the assignment of the signals for the pyridinium protons of (IVa-d) is confirmed by the data from the PMR spectrum of (IVe), which contains an α-picolinium substituent.


IVa-d


IVe

In the case of (IVe) the vibrational rotations of the α-picolinium are restricted so much that this compound only exists in the form of one invertomer, in which the methyl group and the tertiary hydrogen atom are under the screening influence of the tetrahydropyridine system.

The obtained tetrahydropyridine-6-thiolates are stabilized in the form of betaines, and in contrast to the 3,4,5,6-tetrahydro-2(1H)-pyridinethiones [15-17] there is no thione–thiole tautomerism in the thiolates. It is, therefore, the carbonyl-containing quaternized pyridines which are convenient models for investigation of the regio- and stereoselectivity in the reactions leading to the formation of substituted 2(1H)-pyridinethiones from cyanothioacetamide and carbonyl compounds.

EXPERIMENTAL

The IR spectra were recorded on a Specord M-80 instrument for tablets with potassium bromide. The PMR spectra were recorded on a Bruker WM-250 instrument at 250 MHz in DMSO-d_6 with TMS as internal standard. The reactions and the individualities of the compounds were monitored by TLC on Silufol UV-254 plates in the 4:1:1 n-butanol–acetone–hexane system. The 1-styrylpyridinium salts (Ia-d) were obtained by the method in [9, 12], and the cyanothioacetamide (II) was obtained by the method in [20].

The elemental analyses of the compounds for C, H, N, and S (Hal) agree with the calculated data.

4,6-Diaryl-2-cyano-2(1H)-pyridinethiones (IIIa, b). A. A mixture of 5 mmole of the respective compound (Ia, b), 5 mmole of cyanothioacetamide (II), and 10 mmole of ammonium acetate in 15 ml of acetic acid was boiled for 2 h. The reaction mixture was cooled and poured onto 50 g of ice, and the precipitate was separated and washed with water. After twofold recrystallization from nitromethane the yield of (IIIa) amounted to 53%, and the yield of (IIIb) amounted to 48%.

B. A mixture of 0.8 g (2 mmole) of the pyridine-2-thiolate (IVa) and 0.23 g of (3 mmole) of ammonium acetate in 15 ml of acetic acid was boiled for 2 h and poured onto 50 g of ice. The pyridinethione (IIIa) was isolated by analogy with method A with a yield of 0.3 g (49%).

The compounds obtained by methods A and B were identical in their melting points and IR spectra with the 4,6-diaryl-3-cyano-2(1H)-pyridinethiones [13].

2-Hydroxy-2,4-diaryl-3-(1-pyridinio)-5-cyano-3,4-trans-1,2,3,4-tetrahydropyridine-6-thiolates (IV). A. A mixture of 10 mmole of the respective salt (Ia-d), 10 mmole of cyanothioacetamide, and 10 mmole of triethylamine in 20 ml of ethanol was brought to boiling and quickly filtered through a fluted filter. After 6-8 h the precipitate was filtered off, washed with ethanol and with hexane, and dried in air.

B. A moderate stream of hydrogen sulfide was passed through a solution of 1.1 g (3 mmole) of the adduct (VI) and 0.3 ml of triethylamine in 25 ml of ethanol at 20°C for 2 h. The reaction mixture was kept at 20°C for 5 h, and the precipitate was filtered off.

C. A mixture of 10 mmole of the aldehyde (VIIa-c), 10 mmole of the salt (VIIIa-c), 10 mmole of cyanothioacetamide, and 10 mmole of triethylamine in 20-25 ml of ethanol was brought to boiling and quickly filtered through a fluted filter. After 6-8 h the precipitate was filtered off.

3-Benzoyl-1,1-dicyano-2-(3-pyridyl)-3-(1-pyridinio)-1-propanide (VI) ($C_{22}H_{16}N_4O$). To a mixture of 1.84 g (5 mmole) of (Ic) and 0.33 g (5 mmole) of malononitrile in 20 ml of ethanol we added 0.7 ml (5 mmole) of triethylamine. The mixture was stirred at 20°C for 5 h. The precipitate was filtered off and washed with ethanol and with hexane. The yield of (VI) was 1.4 g (80%); mp 147-150°C. IR spectrum, cm^{-1}: 1690 (C=O), 2102 strong (C≡N), 2180 (C≡N). PMR spectrum, δ, ppm: 4.13 (1H, d, 2-H, $^3J_{2,3}$ = 8 Hz); 5.12 (1H, d, 3-H, $^3J_{3,2}$ = 8 Hz); 7.39-7.66 (5H, m, C_6H_5); 7.78 (2H, m, 3H-Py^+); 8.1 (1H, m, 5H-Py^+); 8.28 (2H, m, 4H-Py, 6H-Py); 8.56, 8.62 (2H, m, 2H-Py, 4H, 6H-Py^+); 8.9 (1H, d, 2H-Py^+).

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