

The IR spectra of thin films (IIb-d) and mineral oil suspensions were recorded with a UR-20 spectrometer. The electronic spectra of solutions in ethanol were recorded with a Specord UV-vis spectrophotometer. Benzene solutions of hydrazoic acid were prepared by the method in [8].

2-(2-Furyl)acrylonitriles IIa-h. A 6.7 g (0.03 mole) sample of anhydrous magnesium perchlorate was added to 0.1 mole of the corresponding furylacrolein and 0.11 mole of a benzene solution of hydrazoic acid, after which 0.01 mole of dioxonium perchlorate [5 ml of 1,4-dioxane and 0.01 mole (1.4 ml) of 72% HClO<sub>4</sub> at 35°C (ice bath)] was added dropwise with stirring. The rate of dropwise addition was monitored with respect to the stream of evolved nitrogen, which should be rapid but not too vigorous. When nitrogen evolution ceased (after 1-1.5 h), the mixture was treated with water, and the benzene layer was separated, washed with water, and dried with Na<sub>2</sub>SO<sub>4</sub>. The benzene was removed by distillation at reduced pressure, and the residue was distilled in vacuo. Compounds IIe-h were recrystallized from ethanol.

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#### CONFORMATIONS OF ISOMERIC 2,3,4-TRISUBSTITUTED

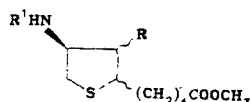
#### TETRAHYDROTHIOPHENES

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The conformations of a number of isomeric 2,3,4-trisubstituted tetrahydrothiophenes were determined by means of angular and temperature dependences of the vicinal proton-proton spin-spin coupling constants (SSCC).

It has previously been shown that differences in the <sup>13</sup>C chemical shifts (CS) of isomeric 2,3,4-trisubstituted tetrahydrothiophenes can be used to determine the structures of the individual isomers and to analyze a mixture of isomers [1]. In the present research we studied the conformations of isomeric 2,3,4-trisubstituted tetrahydrothiophenes I-IV.



I R=OH, R<sup>1</sup>=COC<sub>6</sub>H<sub>5</sub>; II R=OH, R<sup>1</sup>=CONH<sub>2</sub>; III R=OH, R<sup>1</sup>=H·HCl; IV  
R=NHCONH<sub>2</sub>, R<sup>1</sup>=CONH<sub>2</sub>; a 4-r, 3-t, 2-t; b 4-r, 3-t, 2-c; c 4-r, 3-c, 2-c; d 4-r, 3-c, 2-t

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TABLE 1. Vicinal Spin-Spin Coupling Constants ( $^3J$ ) and Differences in the Frequencies of the Most Closely Situated 5-H' and 5-H'' Signals ( $\Delta\delta_{5H',5H''}$ ) for Solutions of the Isomeric I-IV in Deuteropyridine

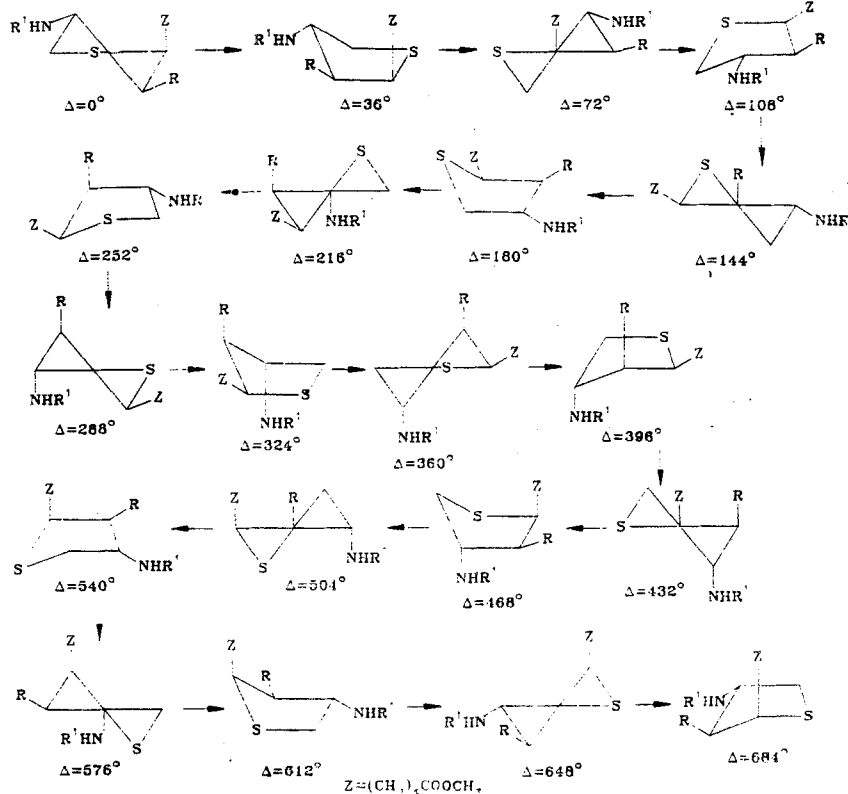
Compound	$^3J$ , Hz				$\Delta\delta_{5H',5H''}$
	2H, 3H	3H, 4H	4H, 5H'	4H, 5H''	
Ia	4,0	4,0	5,7	3,2	41,4
IIa	3,6	3,6	5,2	2,2	61,2
IIIa	5,9	5,9	5,8	5,9	13,0
IVa	—	—	6,0	4,4	30,6
Ib	9,0	8,1	7,2	9,2	22,2
IIb	7,8	7,8	6,7	8,5	31,2
IIIb	9,2	9,2	7,2	10,4	37,2
IVb	8,5	8,5	6,4	8,5	24,6
Ic	3,3	3,3	8,0	10,3	1,0
IIc	3,2	3,2	$\Sigma J_{4-5} = 17,6$		0
IIIc	3,4	3,3	7,2	10,5	28,4
Id	2,0	3,9	$\Sigma J_{4-5} = 16,4$		9,6
IIId	3,8	3,8	$\Sigma J_{4-5} = 14,3$		8,4
IIId	3,9	3,9	6,2	7,8	37,2

A reliable method for the determination of conformations in solution is an analysis of the vicinal proton-proton spin-spin coupling constants (SSCC) by means of their angular dependence [2]. These values for the isomers of I-IV [1, 3-6] are presented in Table 1.

The spectra of Ia-c and IIIa-d were calculated by means of the ITRCALL iteration program in order to determine the true SSCC. The spectra of five-spin systems (the 2-H, 3-H, 4-H, 5-H', and 5-H'' protons) were calculated in each case. The effect of the proton attached to the nitrogen atom and the protons of the closest (to the ring) methylene group of the side chain on the parameters was not taken into account because of the great difference in their chemical shifts ( $\Delta\delta$  is no less than 300 Hz vis-à-vis the maximum J value of 12 Hz). The magnitude of the correction of the constant depends on the difference in the chemical shifts of the most closely situated 5-H' and 5-H'' signals in the spectra. When  $\Delta\delta_{5H',5H''} = 1$  Hz, the maximum correction for  $J_{4H,5H'}$  and  $J_{4H,5H''}$  reaches 3.5 Hz and does not exceed 0.1 Hz when  $\Delta\delta_{5H',5H''} \geq 10$  Hz, i.e., it lies within the limits of the experimental accuracy. Consequently when  $\Delta\delta_{5H',5H''} \geq 10$  Hz it is not necessary to calculate the spectrum in order to refine the values of the vicinal constants, as, for example, for isomers a and b of the investigated compounds. For most isomers c and d  $\Delta\delta_{5H',5H''} < 10$  Hz (Table 1). In this case, a conformational examination was made primarily for those compounds whose spectra were calculated (Ic, IIIc, d). For the remaining compounds (Id, IIc, d) we used the experimentally found, to a first approximation, sums of the vicinal constants with respect to the 4-5 bond, which remain unchanged in the calculation of the spectra.

Because of the complexity of the subjects of the investigation, a massive complex of experimental and computational investigations, including temperature measurements of the constants and processing of them, is required to determine the types of conformations of the five-membered saturated rings and their relative percentages [7]. The procedure can be simplified considerably if the compound exists primarily in one conformation. In this case, the following can be used: a) direct calculation of the torsion angles of the molecule and determination from them of the type of conformation [8]; b) calculation of the constants for all possible conformations and selection of the preferred conformation when its constants coincide with the experimental data. We used the latter method because of its descriptiveness and the possibility of the subsequent use of the results of calculation of the constants of the various conformations of the five-membered ring (see scheme on following page).

The complete cycle of pseudorotation of the five-membered ring includes 20 conformations with a symmetrical skeleton (the substituents were not taken into account in determining the symmetry): 10 envelope conformations ( $C_5$ ) and 10 half-chair conformations ( $C_2$ ) (see the scheme) [9, 10]. Each conformation is characterized by phase angle  $\Delta$ . The phase angle of the complete pseudorotation cycle was assumed to be  $720^\circ$  [11]. The  $C_2$  and  $C_5$  conformations alternate in this chain. The phase angles of adjacent conformations along the cycle differ by  $36^\circ$ . To calculate the vicinal constants of the various conformers we formulated pseudorotation cycles for each of isomers a-d. As the starting conformations (with  $\Delta = 0^\circ$ ) we



selected the  $C_2$  conformations with an equatorial orientation of the substituent attached to the  $C(4)$  atom at the site of maximum puckered character (see Table 2). The pseudorotation cycle for isomer a is shown in the scheme. The pseudorotation cycles for isomers b-d can be obtained from this scheme after appropriate replacement of the configuration of the substituents attached to the  $C(2)$  and  $C(3)$  atoms.

An interrelationship between the dihedral angles of the five-membered ring and the type of conformation was previously found in [8]. By means of these data and the Karplus equation [2] we calculated the vicinal constants for 20 possible conformations of each of the four configurations (a-d) of 2,3,4-trisubstituted tetrahydrothiophene. In the calculations the constant in the Karplus equation was varied from 8 to 12 Hz, i.e., the entire range of possible  $J^\circ$  values was used. It was assumed that  $J^{180^\circ} = 1.35 J^\circ$  [12]. The maximum torsion angle ( $\psi_{\max}$ ) in the ring was selected as being equal to  $50^\circ$ , which is close to the theoretically calculated [9, 10] and experimentally found [11] values for the  $C_2$  and  $C_s$  conformations of the five-membered ring. The results of the calculations are presented in Table 2. In using Table 2 one should take into account the fact that the existence of the five-membered ring in a single conformation is unlikely because of the low conformational energies. Therefore, even in the case of a significant percentage of one of the conformations, there is a contribution of other forms. It was shown that equilibrium between two conformations, the phase angles of which differ by  $360^\circ$  (invert conformations), is characteristic for substituted saturated five-membered rings [11, 13]. The cis constants in such conformations are identical, whereas the trans constants differ significantly [13]. The contribution of the invert conformation with a lower percentage will therefore lead to a change in the trans constants but will not affect the cis constants. It hence follows that preference must be given to the cis constants in the selection of the conformation.

A comparison of the experimental (Table 1) and calculated (Table 2) vicinal constants makes it possible to select the conformation with  $\Delta = 684^\circ$  as the primary conformation for isomers b-d:

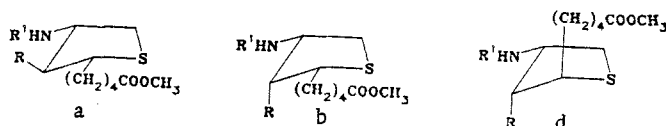
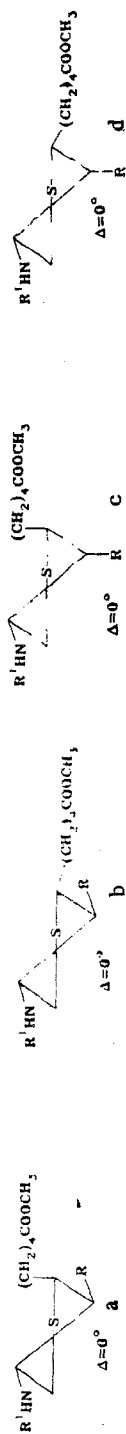


TABLE 2. Calculated  $^3J_{HCCH}$  Values for the Conformations of Isomers a-d of 2,3,4-Trisubstituted Tetrahydrothiophenes



No	$\Delta$ Phase deg	Isomer a					Isomer b					Isomer c					Isomer d				
		$J_{2H, 3H}$	$J_{3H, 4H}$	$J_{4H, 5H^a}$	$J_{4H, 5H^b}$	$J_{4H, 5H^c}$	$J_{2H, 3H}$	$J_{3H, 4H}$	$J_{4H, 5H^a}$	$J_{4H, 5H^b}$	$J_{4H, 5H^c}$	$J_{2H, 3H}$	$J_{3H, 4H}$	$J_{4H, 5H^a}$	$J_{4H, 5H^b}$	$J_{4H, 5H^c}$	$J_{2H, 3H}$	$J_{3H, 4H}$	$J_{4H, 5H^a}$	$J_{4H, 5H^b}$	$J_{4H, 5H^c}$
1	0	4.4-6.7	10.5-15.7	4.4-6.7	9.8-14.7	9.8-14.7	10.5-15.7	4.4-6.7	9.8-14.7	9.8-14.7	9.8-14.7	4.4-6.7	3.3-5.0	4.4-6.7	9.8-14.7	0.3-0.5	3.3-5.0	4.4-6.7	4.4-6.7	9.8-14.7	
2	36	6.0-9.0	10.5-15.7	3.3-5.0	10.5-15.7	10.5-15.7	3.3-5.0	10.5-15.7	10.5-15.7	10.5-15.7	10.5-15.7	3.3-5.0	3.3-5.0	3.3-5.0	10.5-15.7	0	3.3-5.0	3.3-5.0	3.3-5.0	10.5-15.7	
3	72	7.4-11.0	9.8-14.7	3.3-5.0	10.5-15.7	5.6-8.5	9.8-14.7	3.3-5.0	10.5-15.7	10.5-15.7	10.5-15.7	3.3-5.0	3.3-5.0	3.3-5.0	10.5-15.7	0.6-1.0	4.4-6.7	3.3-5.0	3.3-5.0	10.5-15.7	
4	108	8.0-12.0	8.1-12.2	3.3-5.0	10.5-15.7	2.7-4.2	8.1-12.2	3.3-5.0	10.5-15.7	10.5-15.7	10.5-15.7	3.3-5.0	3.3-5.0	3.3-5.0	10.5-15.7	2.7-4.2	6.0-9.0	3.3-5.0	3.3-5.0	10.5-15.7	
5	144	7.4-11.0	5.6-8.5	4.4-6.7	9.8-14.7	0.6-1.0	5.6-8.5	4.4-6.7	9.8-14.7	9.8-14.7	9.8-14.7	4.4-6.7	4.4-6.7	4.4-6.7	9.8-14.7	5.6-8.5	7.4-11.0	4.4-6.7	4.4-6.7	9.8-14.7	
6	180	6.0-9.0	2.7-4.2	6.0-9.0	8.1-12.2	0	2.7-4.2	6.0-9.0	8.1-12.2	8.1-12.2	8.1-12.2	6.0-9.0	6.0-9.0	6.0-9.0	8.1-12.2	8.1-12.2	8.0-12.0	6.0-9.0	6.0-9.0	8.1-12.2	
7	216	4.4-6.7	0.6-1.0	7.4-11.0	5.6-8.5	0.3-0.5	0.6-1.0	7.4-11.0	5.6-8.5	5.6-8.5	5.6-8.5	7.4-11.0	7.4-11.0	7.4-11.0	5.6-8.5	9.8-14.7	7.4-11.0	7.4-11.0	7.4-11.0	5.6-8.5	
8	252	3.3-5.0	0	8.0-12.0	2.7-4.2	0.9-1.4	0.9-1.4	8.0-12.0	2.7-4.2	2.7-4.2	2.7-4.2	8.0-12.0	8.0-12.0	8.0-12.0	2.7-4.2	10.5-15.7	8.0-12.0	8.0-12.0	8.0-12.0	2.7-4.2	
9	288	3.3-5.0	0.3-0.5	7.4-11.0	0.6-1.0	0.9-1.4	0.9-1.4	7.4-11.0	0.6-1.0	0.6-1.0	0.6-1.0	7.4-11.0	7.4-11.0	7.4-11.0	0.6-1.0	10.5-15.7	7.4-11.0	7.4-11.0	7.4-11.0	0.6-1.0	
10	324	3.3-5.0	0.9-1.4	6.0-9.0	0	0.9-1.4	0.9-1.4	6.0-9.0	0	0	0	6.0-9.0	6.0-9.0	6.0-9.0	0	10.5-15.7	6.0-9.0	6.0-9.0	6.0-9.0	0	
11	360	4.4-6.7	0.9-1.4	4.4-6.7	0.3-0.5	0.3-0.5	0.3-0.5	4.4-6.7	0.3-0.5	0.3-0.5	0.3-0.5	4.4-6.7	4.4-6.7	4.4-6.7	0.3-0.5	9.8-14.7	4.4-6.7	4.4-6.7	4.4-6.7	0.3-0.5	
12	396	6.0-9.0	0.9-1.4	3.3-5.0	0.9-1.4	0	0.9-1.4	3.3-5.0	0.9-1.4	0.9-1.4	0.9-1.4	3.3-5.0	3.3-5.0	3.3-5.0	0.9-1.4	8.1-12.2	3.3-5.0	3.3-5.0	3.3-5.0	0.9-1.4	
13	432	7.4-11.0	0.3-0.5	3.3-5.0	0.9-1.4	0.6-1.0	0.3-0.5	3.3-5.0	0.9-1.4	0.9-1.4	0.9-1.4	3.3-5.0	3.3-5.0	3.3-5.0	0.9-1.4	5.6-8.5	4.4-6.7	4.4-6.7	4.4-6.7	0.9-1.4	
14	468	8.0-12.0	0	3.3-5.0	0.9-1.4	2.7-4.2	0.9-1.4	3.3-5.0	0.9-1.4	0.9-1.4	0.9-1.4	3.3-5.0	3.3-5.0	3.3-5.0	0.9-1.4	2.7-4.2	6.0-9.0	6.0-9.0	6.0-9.0	0.9-1.4	
15	504	7.4-11.0	0.6-1.0	4.4-6.7	0.3-0.5	5.6-8.5	0.6-1.0	4.4-6.7	0.3-0.5	0.3-0.5	0.3-0.5	4.4-6.7	4.4-6.7	4.4-6.7	0.3-0.5	7.4-11.0	4.4-6.7	4.4-6.7	4.4-6.7	0.3-0.5	
16	540	6.0-9.0	2.7-4.2	6.0-9.0	0	8.1-12.2	2.7-4.2	6.0-9.0	0	0	0	6.0-9.0	6.0-9.0	6.0-9.0	0	10.5-15.7	6.0-9.0	6.0-9.0	6.0-9.0	0	
17	576	4.4-6.7	5.6-8.5	7.4-11.0	0.6-1.0	9.8-14.7	5.6-8.5	7.4-11.0	0.6-1.0	0.6-1.0	0.6-1.0	7.4-11.0	7.4-11.0	7.4-11.0	0.6-1.0	9.8-14.7	7.4-11.0	7.4-11.0	7.4-11.0	0.6-1.0	
18	612	3.3-5.0	8.1-12.2	8.0-12.0	2.7-4.2	10.5-15.7	8.1-12.2	8.0-12.0	2.7-4.2	2.7-4.2	2.7-4.2	8.0-12.0	8.0-12.0	8.0-12.0	2.7-4.2	10.5-15.7	8.0-12.0	8.0-12.0	8.0-12.0	2.7-4.2	
19	648	3.3-5.0	9.8-14.7	7.4-11.0	5.6-8.5	10.5-15.7	9.8-14.7	7.4-11.0	5.6-8.5	5.6-8.5	5.6-8.5	7.4-11.0	7.4-11.0	7.4-11.0	5.6-8.5	9.8-14.7	7.4-11.0	7.4-11.0	7.4-11.0	5.6-8.5	
20	684	3.3-5.0	10.5-15.7	6.0-9.0	8.1-12.2	10.5-15.7	10.5-15.7	6.0-9.0	8.1-12.2	8.1-12.2	8.1-12.2	6.0-9.0	6.0-9.0	6.0-9.0	8.1-12.2	10.5-15.7	6.0-9.0	6.0-9.0	6.0-9.0	8.1-12.2	

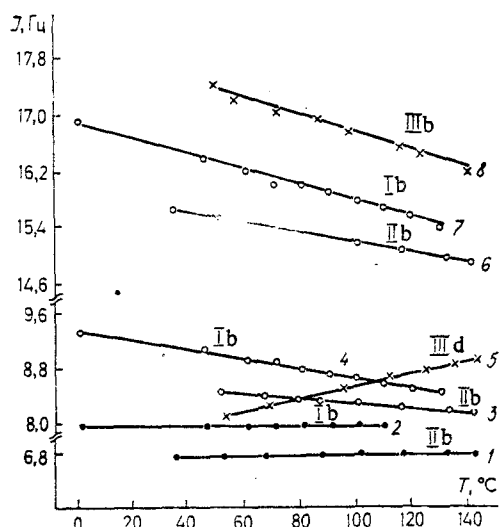
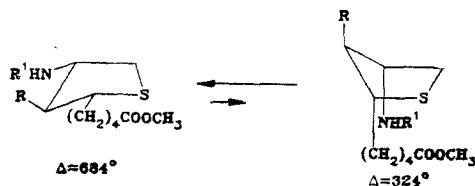


Fig. 1. Temperature dependences of  $J_{4H,5H'}$  (●),  $J_{4H,5H''}$  (○), and the sum of  $J_{2H,3H}$  and  $J_{3H,4H}$  (X).

The experimental values of all of the cis vicinal SSCC ( $J_{2H,3H'}$   $J_{3H,4H}$  = 3.2-3.4 Hz for isomers c, and  $J_{3H,4H}$  = 3.8-3.9 Hz for isomers d; Table 1) and the sums of the vicinal constants with respect to the 4-5 bond ( $\Sigma J_{4-5}$  = 14.0-18.3 Hz; Table 1) are within the limits of the ranges of constants calculated for isomers b-d in this conformation (3.3-5.0 Hz for  $J_{2H,3H}$  and  $J_{3H,4H}$  of isomers c and  $J_{3H,4H}$  of isomers d; 14.1-21.1 Hz for  $\Sigma J_{4-5}$  of isomers b-d; Table 2). The experimental values of the trans vicinal constants for isomers b ( $J_{2H,3H}$  and  $J_{3H,4H}$  = 7.8-9.2 Hz; Table 1) are somewhat smaller than the calculated values (10.5-15.7 Hz; Table 2), whereas in the case of isomers d ( $J_{2H,3H}$  = 2.0-3.9 Hz; Table 1) they are greater than the calculated values (0.9-1.4 Hz; Table 2). The observed deviations of the trans constants can be explained by the 10-20% contribution of conformations with  $\Delta = 324^\circ$ . The increase in these deviations with an increase in the temperature (Fig. 1, curves 3-8) confirms this, since the percentage of the energetically less favorable conformation (with  $\Delta = 324^\circ$ ) increases with an increase in the temperature. As expected, the cis vicinal constants (Fig. 1, curves 1 and 2) remain virtually unchanged when the temperature is changed.

Of the compounds of the a group, only IIa has a preferred conformation. It is intermediate between conformations with a symmetrical skeleton -  $324^\circ < \Delta < 360^\circ$  (see the scheme). A conformation with  $684^\circ < \Delta < 720^\circ$  ( $0^\circ$ ) is in equilibrium with it. Its percentage is 15-25%. In the case of Ia, IIIa, and IVa one cannot select a preferred conformation from Table 2. The populations of the conformations are evidently equalized to a significant degree. The experimental data correspond to an equilibrium of the conformations that are similar to the conformations of IIa: with  $\Delta = 360^\circ$  and  $\Delta = 720^\circ$  for IIIa, and with  $324^\circ < \Delta < 360^\circ$  and  $684^\circ < \Delta < 720^\circ$  ( $0^\circ$ ) for Ia and IVa (see the scheme). For compounds of the a group the percentages of the conformations with  $\Delta = 684-720^\circ$  increase in the order IIa < Ia < IVa < IIIa. For IIIa the percentage of this conformation is 50-60%.

It is interesting to compare the conformational state of 2,3,4-trisubstituted tetrahydrothiophenes and the previously studied [7] 3,4-disubstituted tetrahydrothiophenes and to trace the effect of the substituent attached to the  $C_{(2)}$  atom. For isomers b one observes a shift of the conformational equilibrium to favor a conformation with  $\Delta = 684^\circ$  as compared with the trans-3,4 derivatives [7], in which the populations of the conformations are equalized.



This is explained by an energetically unfavorable 1,3-diaxial interaction in the conformation with  $\Delta = 324^\circ$ , which is absent in trans-3,4-disubstituted tetrahydrothiophenes. An unfavorable 1,3-diaxial interaction also develops in isomers c in the conformation with  $\Delta = 324^\circ$ , but this does not lead to a substantial increase in the percentage of the conformer with  $\Delta = 684^\circ$ , since it is the preferred conformation in the case of cis-3,4-disubstituted

tetrahydrothiophenes. In a and d, in the conformations of which a 1,3-diaxial interaction does not develop, a substituent attached to the C<sub>(2)</sub> atom has virtually no effect on the conformational state. The latter result is in agreement with the well-known fact that the conformational energies of substituents of cyclopentane are small as compared with cyclohexane [14]. In addition, the substituent attached to the C<sub>(2)</sub> atom in the conformations of the investigated compounds is not located at the site of maximum puckered character, where the difference between the axial and equatorial orientations is maximal.

#### EXPERIMENTAL

The vicinal SSCC were obtained with Bruker WP-80 (80 MHz), Wm-250 (250.13 MHz), and WP-200 (200.13 MHz) spectrometers [1, 3-6]. The temperature dependences of the vicinal constants were measured with a WP-200 spectrometer. Traces of oxygen were removed from the samples by bubbling argon through them. The pulse time was 3  $\mu$ sec (30°C). A computer memory of 32 K/1000 Hz was used. The solvent was C<sub>2</sub>D<sub>2</sub>N. The temperature at each point was determined from the dependence of standard samples, viz., ethylene glycol (for high temperatures) and methanol (for low temperatures).

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