

purified by column chromatography (Al_2O_3 , CHCl_3), collecting the colored fraction with R_f 0.85. Yield 0.13 g (55%) colored product.

B. A solution of 0.25 g (0.5 mmole) acid VIIIa in 4 g polyphosphoric acid was heated with stirring on a water bath for 40 min and poured onto ice. The precipitate was separated and dried to give 0.22 g (90%) orange crystals, mp 210°C (from ethanol), R_f 0.85. IR spectrum (cm^{-1}): 1680, 1590, 1035. PMR spectrum (ppm): 3.47 (s, OCH_3), 3.80 (s, OCH_3), 3.87 (s, 3OCH_3), 3.97 (s, OCH_3), 6.17 (s, 1H_{arom}), 6.85 (s, 1H_{arom}), 6.95-7.10 (m, 4H_{arom}), 7.47 (s, 1H_{arom}), 8.18 (s, 1H_{arom}). M^+ 486.

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STEREOCHEMISTRY OF THE IONIC HYDROGENATION OF PENTASUBSTITUTED 4H-THIOPYRANS AND STRUCTURES OF THE THIACYCLOHEXANES OBTAINED FROM THEM

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The structure of 3,5-dimethyl-2,4,6-triphenylthiacyclohexane, obtained as a result of the ionic hydrogenation of the corresponding 4H-thiopyran with the $\text{CF}_3\text{COOH}/\text{HSi}(\text{C}_2\text{H}_5)_3$ couple, was established. It is shown that the stereochemistry of the ionic hydrogenation of 2,3,4,5,6-pentasubstituted 4H-thiopyrans, both those that disproportionate and those that do not disproportionate under the influence of trifluoroacetic acid, is the same.

Pentasubstituted 4H-thiopyrans I-III react differently with trifluoroacetic acid. Thus, thiopyrans II and III undergo disproportionation to the corresponding thiopyrylium salts XII and XIII and thiacyclohexanes VII and VIII like di-, tri-, and tetrasubstituted 4H-thiopyrans IV and V, while sulfide I undergoes quantitative conversion to thiopyrylium trifluoroacetate XI with the participation of air oxygen [1]. 3,5-Dimethyl-2,4,6-triphenylthiacyclohexane (VI), which should be formed in the case of disproportionation of thiopyran I, is obtained only as a result of catalytic (H_2 , Pd/C) or ionic [$\text{CF}_3\text{COOH}/\text{HSi}(\text{C}_2\text{H}_5)_3$] hydrogenation of this compound. The ionic hydrogenation of substituted 4H-thiopyrans can be regarded as a model of their disproportionation under the influence of protic

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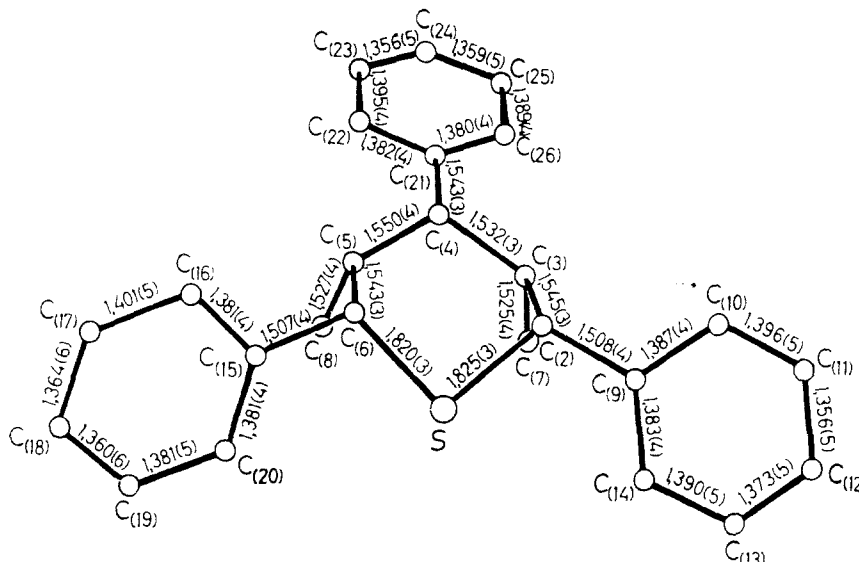
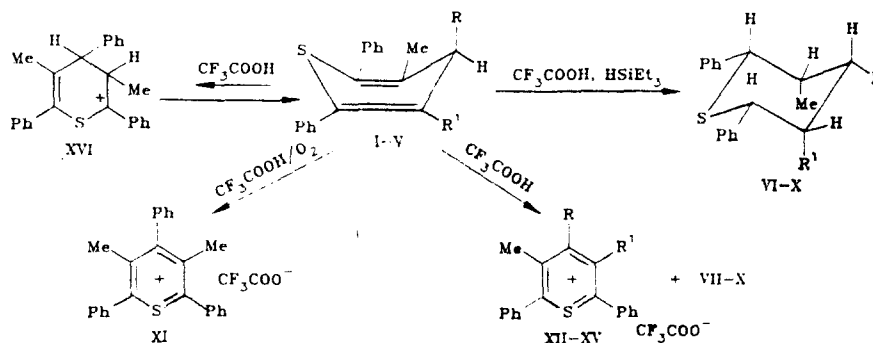


Fig. 1. Geometry of the thiacyclohexane VI molecule.

TABLE 1. ^{13}C NMR Spectra of Thiacyclohexanes VI-X

Compound	Chemical shifts, δ , ppm										
	Carbon atoms in the heteroring					Carbon atoms in the substituents					
	C ₍₂₎	C ₍₃₎	C ₍₄₎	C ₍₅₎	C ₍₆₎	R=Alk at C ₍₄₎	ClI ₃ at C _{(3), C₍₅₎}	C ₆ H ₅ at C _{(2), C₍₆₎}			
								C ₍₁₎	o-	m-	p-
VI	56.10	37.89	52.19	37.89	56.10	—	11.46	141.00	127.63	128.13	126.90
VII	56.14	40.47	42.38	40.47	56.14	18.74	9.72	141.20	127.48	127.97	126.60
VIII	56.49	38.72	50.05	38.72	56.49	25.44	10.07	141.56	127.73	128.17	126.8
							11.86				
IX	54.9	34.2	41.2	34.2	54.9	—	15.8	141.4	127.5	128.0	126.7
X	55.1	41.3	49.1	32.2	48.9	—	6.3	141.9	127.6	128.0	127.4
								140.8	127.2	128.0	127.4

acids. As a result of both reactions, the investigated 4H-thiopyrans I-V form the same isomer of the corresponding thiacyclohexane [2]. Thus a study of the stereochemistry of the ionic hydrogenation of pentasubstituted 4H-thiopyrans, both those that disproportionate (II-V) and those that do not disproportionate (I) in CF_3COOH , is of particular interest.



I, VI R=C₆H₅, R¹=CH₃; II, VII, XII R=R¹=CH₃; III, VIII, XIII R=C₂H₅, R¹=CH₃;
IV, IX, XIV R=H, R¹=CH₃; V, X, XV R=C₆H₅, R¹=H

Pentasubstituted thiacyclohexanes VI-VIII exist in the chair conformation, and in solution in CDCl_3 the conformational equilibrium is shifted virtually completely to favor the conformer with equatorial phenyl (attached to the C₍₂₎ and C₍₆₎ atoms) and axial methyl (attached to the C₍₅₎ and C₍₃₎ atoms) groups [2]. A comparison of the PMR spectra of sulfides VI, IX, and X [3] showed that in thiacyclohexane VI the protons of each of the methyl

substituents (attached to the $C_{(3)}$ or $C_{(5)}$ atom) fall into the region of shielding of only one vicinal phenyl ring (attached to the $C_{(2)}$ or $C_{(6)}$ atom). On the basis of this we concluded that the phenyl group in the 4 position in sulfide VI is axially oriented. It followed from the results obtained that, in the ionic hydrogenation of thiopyran I, the three-dimensional structure of which is known [4], the approach of the hydrogenating agents [CF_3COOH and $HSi(C_2H_5)_3$] to the double bonds of the heteroring is realized from the sterically hindered "frontal side." This fact may serve as one of the reasons why thiopyran I does not undergo disproportionation under the influence of CF_3COOH , since hydrogenation of the heteroring by the CF_3COOH -starting 4H-thiopyran couple should occur in this case [3].

To ascertain the stereochemistry of the ionic hydrogenation [$CF_3COOH/HSi(C_2H_5)_3$] and the disproportionation (in CF_3COOH) of thiopyrans II and III, the three-dimensional structures of which have been established [4], it was necessary to analyze, in addition to the PMR spectra, the ^{13}C NMR spectra of thiacyclohexanes VII and VIII obtained in these reactions. In the spectrum of pentasubstituted thiacyclohexane VII the signal of the heteroring $C_{(2)}$ and $C_{(6)}$ atoms is shifted to weak field as compared with those in the spectrum of tetrasubstituted thiacyclohexane IX (Table 1), which constituted evidence for an equatorial orientation of the methyl group in the 4 position in sulfide VII. A comparison of the chemical shifts of the carbon atoms of the methyl substituents in the spectra of sulfide VII (Table 1) and methyl-substituted cyclohexanes [5] and thiacyclohexanes [6, 7] also shows that the methyl group attached to the $C_{(4)}$ atom is equatorial, while the methyl substituents in the 3 and 5 positions are axial. As in the spectrum of thiacyclohexane VII, in the spectra of pentasubstituted thiacyclohexanes VI and VIII, which have a phenyl and ethyl group, respectively, in the 4 position of the heteroring, one observes a weak-field shift of the signal of the $C_{(2)}$ and $C_{(6)}$ atoms as compared with the corresponding signal in the spectrum of sulfide IX; the shifts for thiacyclohexanes VI-VIII are close and amount to 1.42 ± 0.18 ppm (Table 1). This indicates an identical orientation of the substituents attached to the $C_{(4)}$ atom in sulfides VI-VIII. Thus, according to the ^{13}C NMR spectral data, in pentasubstituted thiacyclohexanes VI-VIII both the alkyl and phenyl substituents in the 4 position are oriented equatorially.

To resolve the contradiction that arises in interpreting the data from the PMR [3] and ^{13}C NMR spectra of thiacyclohexane VI we carried out an x-ray diffraction analysis of this sulfide. It was established that the thiacyclohexane VI molecule exists in a chair conformation with an equatorial orientation of the phenyl groups (attached to the $C_{(2)}$, $C_{(4)}$, and $C_{(6)}$ atoms) and an axial orientation of the methyl groups (attached to the $C_{(3)}$ and $C_{(5)}$ atoms) (Fig. 1). The S and $C_{(4)}$ atoms deviate from the $C_{(2)}C_{(3)}C_{(5)}C_{(6)}$ plane (A), which is realized with an accuracy of 0.007 Å, by 0.988(1) Å and $-0.654(2)$ Å, respectively (Table 2). The dihedral angles between plane A and angles $C_{(2)}SC_{(6)}$ and $C_{(3)}C_{(4)}C_{(5)}$ are $54.0(1)^\circ$ and $52.5(1)^\circ$. The endocyclic torsion angles [57.5 - $63.7(3)^\circ$] somewhat exceed those found in a low-temperature x-ray diffraction study of cyclohexane [8], which has an ideal chair conformation, as well as in the sulfide IX molecule (52.8° , 58.3° , 52.8°) [2]. The 2,6-phenyl substituents are turned relative to plane A of the middle part of the heteroring by $86.1(1)^\circ$ and $63.2(1)^\circ$, respectively. The dihedral angle between plane A and the plane of the phenyl substituent in the 4 position is only $13.5(1)^\circ$. Each vicinal pair of substituents in the sulfide VI molecule exists in a gauche conformation: the exocyclic torsion angles $C_{(7)}C_{(3)}C_{(2)}C_{(9)}$ [$-58.1(3)^\circ$], $C_{(7)}C_{(3)}C_{(4)}C_{(21)}$ [$61.1(3)^\circ$], $C_{(8)}C_{(5)}C_{(4)}C_{(21)}$ [$-61.0(3)^\circ$], and $C_{(8)}C_{(5)}C_{(6)}C_{(15)}$ [$56.8(3)^\circ$] are close to 60° . Steric hindrance that arises due to 1,3-diaxial interaction of methyl groups attached to the $C_{(3)}$ and $C_{(5)}$ atoms is resolved due to an increase in bond angle $C_{(3)}C_{(4)}C_{(5)}$ to $115.3(2)^\circ$ as compared with the tetrahedral angle (109.5°). However, the $C_{(7)}-C_{(8)}$ distance, which is equal to $3.266(4)$ Å, remains substantially lower than twice van der Waals radius of methyl group (4.0 Å [9, p. 106]), which constitutes evidence for retention of steric strain in molecule.

The S—C bond lengths have the usual values and coincide with those previously found [2, 10]. The $C_{(2)}SC_{(6)}$ bond angle is smaller than that in thiacyclohexane IX [$97.9(4)^\circ$] [2], cis-2-trans-6-diphenyl-cis-3-ethyltetrahydrothiopyran-4-ol (99.8°) [10], or 2,2,6,6-tetramethyl-4[e]-phenyltetrahydrothiopyran-4[a]-ol (105.6°) [10]. The lengths of the exocyclic $C_{(2)}-C_{(9)}$ and $C_{(6)}-C_{(15)}$ bonds actually coincide with the standard value for the $C_{sp^3}-C_{sp^2}$ bond, which is equal to 1.510 Å [11], while the length of the $C_{(4)}-C_{(21)}$ bond is formally of the same type as the $C_{(2)}-C_{(9)}$ and $C_{(6)}-C_{(15)}$ bonds; however, it is somewhat greater than the lengths of the latter.

The bond angles at the key atoms of the phenyl substituents $C_{(10)}C_{(9)}C_{(14)}$, $C_{(16)}C_{(15)}C_{(20)}$, and $C_{(22)}C_{(21)}C_{(26)}$ are decreased as compared with the ideal value for the bond angles in benzene (120°). This decrease constitutes evidence for the electron-donor character of the 2, 4, and 6 positions of the tetrahydrothiopyran ring [12]. The same change in the bond angles in the phenyl rings is also observed in structure IX [2].

The inequality of the exocyclic bond angles at the $C_{(2)}$ and $C_{(6)}$ atoms ($C_{(3)}C_{(2)}C_{(9)}$ and $SC_{(2)}C_{(9)}$, as well as angles $C_{(5)}C_{(6)}C_{(15)}$ and $SC_{(6)}C_{(15)}$) is customary for α -substituted S,O-containing heterocycles [2, 10, 13].

TABLE 2. Bond Angles in the Sulfide VI Molecule

Angle	ω°
$C_{(2)}SC_{(6)}$	95,9(1)
$SC_{(2)}C_{(5)}$	111,5(2)
$SC_{(2)}C_{(9)}$	110,1(2)
$C_{(3)}C_{(2)}C_{(9)}$	114,4(2)
$C_{(2)}C_{(3)}C_{(4)}$	110,4(2)
$C_{(2)}C_{(3)}C_{(7)}$	113,1(2)
$C_{(4)}C_{(3)}C_{(7)}$	115,6(2)
$C_{(3)}C_{(4)}C_{(5)}$	115,3(2)
$C_{(3)}C_{(4)}C_{(21)}$	113,4(2)
$C_{(5)}C_{(4)}C_{(21)}$	113,2(2)
$C_{(4)}C_{(5)}C_{(6)}$	111,1(2)
$C_{(4)}C_{(5)}C_{(8)}$	116,0(2)
$C_{(6)}C_{(5)}C_{(8)}$	111,9(2)
$C_{(5)}C_{(6)}S$	111,8(2)
$SC_{(6)}C_{(15)}$	111,4(2)
$C_{(5)}C_{(6)}C_{(15)}$	113,0(2)
$C_{(12)}C_{(9)}C_{(10)}$	123,2(2)
$C_{(2)}C_{(9)}C_{(14)}$	119,1(2)
$C_{(10)}C_{(9)}C_{(14)}$	117,6(3)
$C_{(9)}C_{(10)}C_{(11)}$	121,6(3)
$C_{(10)}C_{(11)}C_{(12)}$	119,4(3)
$C_{(11)}C_{(12)}C_{(13)}$	120,4(3)
$C_{(12)}C_{(13)}C_{(14)}$	120,3(3)
$C_{(13)}C_{(14)}C_{(9)}$	120,7(3)
$C_{(6)}C_{(15)}C_{(16)}$	123,7(3)
$C_{(6)}C_{(15)}C_{(20)}$	118,3(3)
$C_{(16)}C_{(15)}C_{(20)}$	118,0(3)
$C_{(15)}C_{(16)}C_{(17)}$	119,7(3)
$C_{(16)}C_{(17)}C_{(18)}$	121,0(4)
$C_{(17)}C_{(18)}C_{(19)}$	119,8(4)
$C_{(18)}C_{(19)}C_{(20)}$	119,9(4)
$C_{(19)}C_{(20)}C_{(15)}$	121,5(3)
$C_{(4)}C_{(21)}C_{(22)}$	121,4(2)
$C_{(4)}C_{(21)}C_{(26)}$	121,8(2)
$C_{(22)}C_{(21)}C_{(26)}$	116,6(2)
$C_{(21)}C_{(22)}C_{(23)}$	121,3(3)
$C_{(22)}C_{(23)}C_{(24)}$	120,6(3)
$C_{(23)}C_{(24)}C_{(25)}$	119,4(3)
$C_{(24)}C_{(25)}C_{(26)}$	120,2(3)
$C_{(25)}C_{(26)}C_{(21)}$	121,9(3)

TABLE 3. Coordinates of the Atoms ($\times 10^4$, $\times 10^3$ for H) and Their Equivalent Isotropic (isotropic for the H atoms) Temperature Factors of the Sulfide VI Molecule

Atom	x	y	z	B_{iso}^{eq}/B_{iso} , \AA^2
S	1611(1)	666(1)	1723(1)	4,37(2)
$C_{(2)}$	2895(2)	-313(2)	1547(1)	4,03(7)
$C_{(5)}$	2370(2)	-1520(2)	1124(1)	3,97(7)
$C_{(4)}$	1703(2)	-2310(2)	1622(1)	3,84(7)
$C_{(3)}$	577(2)	-1669(2)	1869(1)	4,02(7)
$C_{(6)}$	1015(3)	-457(2)	2310(1)	4,18(7)
$C_{(7)}$	1610(3)	-1295(2)	306(2)	4,48(8)
$C_{(8)}$	-639(3)	-1482(2)	1242(2)	4,59(8)
$C_{(9)}$	3751(2)	431(2)	1162(2)	4,40(8)
$C_{(10)}$	3295(3)	1283(3)	587(2)	5,42(9)
$C_{(11)}$	4110(3)	1918(3)	226(2)	6,5(1)
$C_{(12)}$	5401(3)	1701(3)	446(2)	7,1(1)
$C_{(13)}$	5879(3)	884(3)	1015(2)	7,2(1)
$C_{(14)}$	5060(3)	234(3)	1372(2)	5,88(9)
$C_{(15)}$	3(3)	104(2)	2660(2)	4,72(8)
$C_{(16)}$	-618(3)	1205(3)	2416(2)	6,5(1)
$C_{(17)}$	-1555(4)	1649(3)	2771(2)	8,3(1)
$C_{(18)}$	-1859(4)	1018(4)	3355(2)	8,4(1)
$C_{(19)}$	-1253(4)	-67(4)	3601(2)	8,7(2)
$C_{(20)}$	-318(3)	-512(3)	3261(2)	7,0(1)
$C_{(21)}$	1381(2)	-3635(2)	1309(1)	4,13(7)
$C_{(22)}$	443(3)	-4335(2)	1517(2)	5,14(8)
$C_{(23)}$	207(3)	-5558(3)	1269(2)	6,4(1)
$C_{(24)}$	900(3)	-6095(3)	818(2)	6,4(1)
$C_{(25)}$	1832(3)	-5430(3)	606(2)	6,6(1)
$C_{(26)}$	2075(3)	-4213(2)	854(2)	5,51(9)
$H_{(2)}$	335(2)	-52(2)	204(1)	3,4(5)
$H_{(3)}$	315(2)	-196(2)	107(1)	4,2(5)
$H_{(4)}$	232(2)	-241(2)	210(1)	3,3(5)
$H_{(5)}$	38(2)	-221(2)	226(1)	3,2(4)
$H_{(6)}$	172(2)	-63(2)	268(1)	3,8(5)
$H_{(7.1)}$	97(2)	-67(2)	29(1)	4,9(6)
$H_{(7.2)}$	216(2)	-99(2)	-3(1)	5,2(6)
$H_{(7.3)}$	132(3)	-205(2)	16(1)	6,4(6)
$H_{(8.1)}$	-62(2)	-77(2)	97(1)	5,3(6)
$H_{(8.2)}$	-134(2)	-136(2)	148(1)	5,4(6)
$H_{(8.3)}$	-83(2)	-221(2)	87(1)	5,4(6)
$H_{(10)}$	244(2)	139(3)	47(1)	6,1(6)
$H_{(11)}$	369(3)	248(3)	-24(2)	8,4(8)
$H_{(12)}$	589(3)	221(3)	15(2)	9,6(9)
$H_{(13)}$	655(7)	75(7)	111(4)	17(3)
$H_{(14)}$	535(2)	-37(2)	177(1)	5,5(6)
$H_{(16)}$	-51(3)	169(3)	194(2)	9,0(9)
$H_{(17)}$	-197(3)	234(3)	254(2)	9,1(8)
$H_{(18)}$	-245(3)	123(3)	363(2)	9,4(9)
$H_{(19)}$	-143(3)	-57(3)	404(2)	8,2(8)
$H_{(20)}$	12(3)	-129(3)	342(2)	9,6(9)
$H_{(22)}$	-1(2)	-403(2)	186(1)	4,3(5)
$H_{(23)}$	-42(2)	-595(2)	143(1)	6,1(7)
$H_{(24)}$	74(3)	-685(2)	65(2)	6,5(7)
$H_{(25)}$	237(3)	-577(3)	27(2)	9,8(9)
$H_{(26)}$	271(3)	-374(3)	71(2)	7,4(7)

Thus, from the results of an x-ray analysis of penta-substituted thiacyclohexane VI it becomes clear that, in contrast to 3a-methyl-2e,4e,6e-triphenylthiacyclohexane X, in which the protons of the methyl substituent fall into the region of shielding of the two equatorially oriented phenyl rings in the 2 and 4 positions [3], in thiacyclohexane VI the protons of the corresponding methyl group in the 3 or 5 position are shielded only by the aromatic ring attached to the $C_{(2)}$ or $C_{(6)}$ atom, since the phenyl ring attached to the $C_{(4)}$ atom, being equatorially oriented, is turned relative to heteroring plane A by an angle (13.5°) such that the protons of the methyl groups under consideration do not fall into the region of its shielding. This also led to the erroneous conclusion relative to the

orientation of the phenyl substituent in the 4 position in sulfide VI that was drawn on the basis of PMR spectral data [3].

The results obtained provide evidence that the stereochemistry of the ionic hydrogenation of pentasubstituted 4H-thiopyrans I-III and of the disproportionation of thiopyrans II and III under the influence of CF_3COOH is the same. The corresponding hydrogenating agents [$\text{CF}_3\text{COOH}-\text{HSi}(\text{C}_2\text{H}_5)_3$ or CF_3COOH -starting 4H-thiopyran molecule] approach the double bonds of the thiopyran ring from the "rear." As previously assumed [1], the differences in the behavior of thiopyrans I and II and III with trifluoroacetic acid are most likely associated with a decrease in the hydride lability of the hydrogen atom attached to the $\text{C}_{(4)}$ atom of thiopyran I as compared with that in thiopyrans II and III. As in the case of thiopyrans II and III, protonation of the double bonds of the heteroring occurs in the reaction of 4H-thiopyran I with trifluoroacetic acid, and the corresponding carbonium ion XVI develops [1]. However, whereas stabilization of the resulting carbonium ion in the disproportionation of 4H-thiopyrans, particularly sulfides II and III, is realized due to hydride transfer from the starting thiopyran, carbonium ion XVI is stabilized due to the ejection of a proton. As a result, thiopyran I is converted to thiopyrylium trifluoroacetate XI via a competitive (with disproportionation) mechanism with the participation of oxygen.

EXPERIMENTAL

The ^{13}C NMR spectra of solutions of thiacyclohexanes VI-X in CDCl_3 were obtained with a Varian FT-80A spectrometer (20 MHz) at 30°C with hexamethyldisiloxane (HMDS) as the internal standard; the shift relative to tetramethylsilane (TMS) was 1.91 ppm. Thiacyclohexanes VI-X were obtained by the method in [1].

X-Ray Diffraction Analysis of Thiacyclohexane VI. The crystals of VI were monoclinic and had the following parameters: $a = 10.775(1)$, $b = 10.771(1)$, $c = 17.984(2)$ Å, $\beta = 103.67(1)^\circ$, $V = 2028.1(5)$ Å³, $Z = 4$, $d_{\text{calc}} = 1.174$ g/cm³, space group $\text{P}2_1/\text{n}$. The cell parameters and intensities of 1945 independent reflections with $F^2 \geq 3\sigma$ were measured with a Hilger-Watts Y/200 automatic four-circle diffractometer (20°C , $\lambda_{\text{Mo K}\alpha}$, graphite monochromator, $\theta/2\theta$ scanning, $\theta \leq 30^\circ$). The structure was decoded by the direct method by means of the MULTAN program and was refined by the total-matrix method of least squares, initially within the isotropic approximation and then within the anisotropic approximation. All of the H atoms were revealed objectively in differential synthesis and were included in the refinement within the isotropic approximation. The final R factor was 0.038 ($R_w = 0.047$). All of the calculations were made with an Eclipse S/200 computer by means of the INEXTL program [14]. The coordinates of the atoms and their temperature factors are presented in Table 3.

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