CONVERSION OF trans, trans-1-METHOXY-3, 5-DIARYL-2-OXABICYCL0[4.4.0]DEC-

3-ENES INTO HEMIDITHIOACETALS AND 4H-THIOPYRANS.

STRUCTURE OF trans, trans-1-MERCAPTO-3-PHENYL-5-(4-METHOXYPHENYL)-2-THIABICYCL0[4.4.0]DEC-3-ENE

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The conversion of trans, trans-1-methoxy-3,5-diaryl-2-oxabicyclo[4.4.0]dec-3-enes into trans, trans-1-mercapto-3,5-diaryl-thiabicyclo[4.4.0]dec-3-enes or 2,4-diaryl-5,6-tetramethylene-4H-thiopyrans, which takes place when hydrogen sulfide is reacted with the acetals in acetic acid with the use of gaseous hydrogen chloride as a catalyst, has been described. A mechanism for the reaction has been proposed. The structure and confirmations of the molecules have been determined by x-ray diffraction analysis and <sup>13</sup>C NMR.

According to the data in [1], the reactions of 1,3-diaryl-3-(2-oxocyclohexyl)-1-propanones with hydrogen sulfide in methanol in the presence of hydrogen chloride result in the formation of either trans,trans-3,5-diaryl-2-thiabicyclo[4.4.0]dec-3-enes or trans,trans-1-methoxy-3,5-diaryl-2-oxabicyclo[4.4.0]dec-3-enes (I). In the latter case, an interaction with one of the nucleophiles, i.e., with methanol, takes place. It was noted that the conversion of oxygen analogs of I into sulfur analogs of type II is not observed in methanol. At the same time, it is known that acetals are split under the action of hydrogen chloride. In this context it seemed to be of interest to study the reactions of bicyclic acetals Ia-c with hydrogen sulfide and hydrogen chloride in a polar solvent.

For this purpose we studied the reactions of trans, trans-acetals Ia-c with hydrogen sulfide in acetic acid under the conditions of acid catalysis. It was established that the reaction of hydrogen chloride with a suspension of trans, trans-acetals Ia-c in acetic acid which had been saturated to the limit with hydrogen sulfide at 10-15°C results in the formation of trans, trans-hemithioacetals IVa and IVc. If the reaction mixture is subjected to the simultaneous action of hydrogen sulfide and hydrogen chloride, 4H-thiopyrans IIIb and IIIc are recovered as the reaction products. Thioacetals IIa-c were not detected.

The opening of the acetal ring in compounds Ia-c should produce a carbonium ion (A), which is structurally similar to the protonated form of 1,3-diaryl-3-(2-oxocyclohexyl)-1propanones. Since the equatorial attack of ions of such a type is sterically hindered, as was shown in [1], the preferential axial attack of ion A should produce intermediate B. The absence of trans, trans-thioacetals IIa-c in the reaction products attests to the fact that intermediate C with a configuration which is most favorable for the intramolecular interaction of the mercapto group with the carbonyl group clearly does not form. The intramolecular interaction of the mercapto and carbonyl groups which occupy axial and equatorial positions in hemithioacetal B is impeded by the rigidity of the acyclic chain, and the formation of cis, trans isomers of type II is also not observed. The elimination of methanol from intermediate B gives carbonium ion D and then either 4H-thiopyrans IIIb and IIIc, if the concentration of hydrogen sulfide is small, of trans, trans-hemidithioacetals IVa and IVc via gem-dithiol E.

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a. c  $R = C_6 H_5$ , b  $R = C_6 H_4 OCH_3 - 4$ ; a, b  $R^1 = C_6 H_5$ , c  $R^1 = C_6 H_4 OCH_3 - 4$ 

A necessary condition for the formation of gem-diothiols of type E and then of trans, trans compounds IVa and c as intermediates is the limiting saturation of the reaction mixture at 10-15°C with hydrogen sulfide. When hydrogen sulfide and hydrogen chloride are simultaneously reacted with acetals Ib and Ic, the dissolution of the original compounds Ib and Ic and the precipitation of 4H-thiopyrans IIIb and c are observed after 30-40 min.

When there is a shortage of hydrogen sulfide in the mixture, ion A can be subjected to the attack of another nucleophile, viz., water, which is evolved when 4H-thiopyrans of type III are formed. This results in the formation of the products of the intramolecular cyclodehydration of 1,5-diketones of type V, i.e., 2,4-diarylbicyclo[3.3.1]non-2-en-9-ones of type VI.

The intramolecular cyclodehydration process  $E \rightarrow IVa,c$  is stereospecific and results in the formation of substances with a trans,trans configuration. The trans-diequatorial arrangement of the interacting parts in gem-dithiol E is most favorable for cyclization. This is consistent with the results in [1].



When the experiment is conducted in the absence of acetic anhydride, a small quantity (9.5%) of compound VIc is recovered along with 4H-thiopyran IIIc.

For the purpose of confirming the proposed mechanism for the conversion of trans, transacetals of type I into hemidithioacetals of type IV, we determined the crystal and molecular structure of 1-mercapto-3-phenyl-5-(4-methoxyphenyl)-2-thiabicyclo[4.4.0]dec-3-ene (IVc). The crystals of compound IVc are monoclinic:  $\alpha = 12.575(1)$ , b = 10.2942(8), c = 16.425(1)Å,  $\beta = 115.71(1)^{\circ}$ , V = 1915.7 Å<sup>3</sup>, M = 368,  $d_{cal} = 1.28$  g/cm<sup>3</sup>, z = 4, space group  $P2_{1/c}$ ,  $\mu(Cu K\alpha) = 24.7$  cm<sup>-1</sup>. The coordinates of the atoms are listed in Table 1, and the equations of the planar fragments in the molecule and the deviations of the atoms from them are given in Table 2. The stereochemistry of the molecule with the principal bond lengths and bond angles is shown in Fig. 1, and the torsion angles are presented in Fig. 2.

The geometry of dihydrothiopyran ring A is close to that found in the molecules of trans, trans-1-methoxy-3-(4-methoxypheny1)-5-pheny1-2-thiabicyclo[4.4.0]dec-3-ene [2] and cis-3,5-dipheny1-2-thiabicyclo[4.4.0]decene- $\Delta^{1,6}$  [3]. The geometry of the methoxy group is close







Fig. 1. Bond lengths and bond angles in compound IVc.  $S_{(1)}-C_{(1)}=1.835(4)$ ;  $S_{(1)}C_{(1)}S_{(2)}=108.6(2)$ ;  $S_{(1)}C_{(1)}C_{(10)}=109.4(3)$ ;  $S_{(2)}C_{(1)}C_{(10)}=105.6(3)$ ;  $C_{(1)}-C_{(6)}=1.546(6)$ ;  $S_{(1)}C_{(1)}C_{(6)}=112.7(3)$ .

Fig. 2. Torsion angles in compound IVc.

to that found in the structure of 4-methoxybenzoic acid [4], where O-C(Me) = 1.443, O-C(Ph) = 1.360 Å, and the C-O-C angle is equal to 116.8°. The length of the  $S_{(1)}-C_{(1)}$  bond in the mercapto group, which is equal to 1.835(4) Å, does not coincide with the standard value of 1.817(5) Å [5].

The bicyclic system has trans annelation. Dihydrothiopyran ring A has a "half-chair" conformation, the  $C_{(1)}$  and  $C_{(6)}$  atoms deviate in opposite directions from the planar  $S_{(2)}C_{(3)}-C_{(4)}C_{(5)}$  fragment; cyclohexane ring B has a "chair" conformation. Phenyl rings C (in position 3) and D (in position 5) are planar. The dihedral angles between rings C and D and the planar fragment of the heterocycle are 46.7 and 99.4°, respectively. Phenyl substituent D is found in a pseudoequatorial position.

l-Mercapto-3,5-diphenyl-2-thiabicyclo[4.4.0]dec-3-ene (IVa), like hemidithioacetal IVc, has a trans, trans configuration, which was established on the basis of an analysis of their <sup>13</sup>C NMR spectra\* (see Experimental section).

4H-Thiopyrans IIIb and IIIc were identified on the basis of the characteristic signals of the vinyl and 4-H protons in the PMR spectra [6].

## EXPERIMENTAL

The unit-cell parameters and the intensities of 1772 independent reflections with I > 3 $\sigma$  were measured on a Hilger-Watts automatic four-circle diffractometer, which was controlled by a PDP 8/I computer, in Cu K $\alpha$  radiation with a graphite monochromator,  $\theta/2\theta$  scanning, and I  $\leq \theta \leq 57^{\circ}$ . The structure was solved by the direct method according to the MULTAN program and refined by the least-squares method in the full-matrix anisotropic approximation. All the hydrogen atoms were revealed by a difference synthesis and taken into account in the concluding steps of the refinement with fixed positional and thermal parameters (it was assumed that  $B_{150} = 5.0$  Å<sup>2</sup>). The final values of the R factors were R = 0.0514 and R<sub>w</sub> = 0.0641.

<sup>\*</sup>A detailed discussion of the <sup>13</sup>C NMR spectra of hemidithioacetals of type IV and related heterocyclic systems will be presented in a separate report.

TABLE 1. Coordinates of Atoms (×10<sup>4</sup>)

a an	]				1		T	
Atom	×	y	z	Atom	*	IJ	2	
Atom $S_{(1)}$ $S_{(2)}$ O $C_{(3)}$ $C_{(4)}$ $C_{(4)}$ $C_{(4)}$ $C_{(6)}$ $C_{(3)}$ $C_{(4)}$ $C_{(1)}$	* 4214 (1) 5612 (1) -548 (2) 4231 (3) 5319 (3) 4245 (3) 3096 (3) 3167 (3) 2023 (3) 2050 (3) 3097 (4) 4238 (3) 6393 (3) 7451 (3) 8442 (4) 8392 (4) 7346 (4) 6362 (3) 2130 (3)	y 3376 (1) 1777 (1) 2234 (3) 1866 (3) 2820 (4) 3203 (4) 3203 (4) 3203 (4) 1585 (4) 445 (4) 547 (4) 695 (3) 3092 (3) 3491 (4) 3154 (4) 2964 (3)	z   8061 (1)   9676 (1)   10602 (2)   8661 (2)   10261 (3)   9475 (2)   8893 (2)   8042 (3)   7424 (2)   1260 (2)   1260 (2)   12872 (3)   12881 (3)   12090 (3)   9784 (2)	Atom $H_{(8)}$ $H_{(4)}$ $H_{(4)}$ $H_{(6)}$ $H_{(7)}$ $H_{(7')}$ $H_{(8)}$ $H_{(8')}$ $H_{(9')}$ $H_{(10')}$ $H_{(10')}$ $H_{(12)}$ $H_{(12)}$ $H_{(13)}$ $H_{(14)}$ $H_{(18)}$ $H_{(18)}$ $H_{(18)}$	x 3187 4195 2891 3293 1370 1863 1308 2118 2988 3137 4903 4367 7490 9200 9114 7298 5592 1075	y 3518 3772 3689 956 1435 2401 442 - 383 1325 - 253 805 - 113 3556 3903 3622 3059 2685 4220	2 7729 10735 9074 9267 8225 7677 6883 7791 6883 7791 6831 6898 7907 8451 10667 12027 13453 13470 12106 9067 0 650	
C <sub>(16)</sub> C <sub>(17)</sub>	6362 (3) 2129 (3)	2945 (4) 2694 (3)	12090 (3) 9784 (2)	$H_{(18)}$ $H_{(19)}$	1075 398	4220 3941	9067 9583	
$\begin{array}{c} C_{(18)} \\ C_{(19)} \\ C_{(20)} \\ C_{(21)} \\ C_{(22)} \\ C_{(22)} \end{array}$	$\begin{array}{c} 1154 (3) \\ 291 (3) \\ 368 (3) \\ -1335 (3) \\ 2210 (3) \\ 506 (4) \end{array}$	3500 (4) 3334 (4) 2331 (4) 1520 (4) 1715 (4)	9499 (3) 9789 (3) 10369 (2) 10667 (2) 10378 (2)	H (21) H (22) H (23) H (23') H (23")	$1409 \\ 2911 \\ -1389 \\ 56 \\ -442$	796 1123 1111 1049 350	11096 10607 11111 11771 10763	
(23)	- 050 (4)	1123 (4)	11093 (3)					

\*The numbers of the H atoms coincide with the numbers of the corresponding nonhydrogen atoms (with single and double prime signs for the second and third H atoms), and the hydrogen atom of the mercapto group is denoted by H(S).

The <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on a Varian FT-80A spectrometer (80 MHz) with HMDS (for <sup>1</sup>H) and CDCl<sub>3</sub> (for <sup>13</sup>C) as internal references. The course of the reactions was monitored by TLC on Silufol UV-254 plates with a 6:1 hexane-ether mixture as the eluent.

The original acetals Ia-c were obtained in analogy to [7].

Conversion of trans, trans-1-Methoxy-3, 5-diary1-2-oxabicyclo[4.4.0]-dec-3-enes (Ia, c) into trans, trans-1-Mercapto-3-pheny1-5-(4-methoxypheny1)-2-thiabicyclo[4.4.0]dec-3-ene (IVc) and trans, trans-1-Mercapto-3,5-dipheny1-2-thiabicyclo[4.4.0]dec-3-ene (IVa). A suspension of 3.6 g (15 mmole) of acetal Ic in 40 ml of glacial acetic acid (with an addition of 2 ml of acetic anhydride) is saturated with hydrogen sulfide for 1.5 h at 10-15°C and then with a mixture of hydrogen sulfide and hydrogen chloride for 1 h. After 0.5 h has elapsed from the beginning of the passage of HCl, initial acetal Ic is dissolved, and crystallization of reaction product IVc is observed. The mixture is left to stand for 17 h in a refrigerator, and then compound IVc is filtered out, washed with water, and dried in air. This gives 2.05 g of hemidithioacetal IVc with  $T_m = 139.5-140.5^\circ$  (from petroleum ether). The mother solution is poured onto ice, and another 1.5 g of IVc are recovered; the total yield is 94%. <sup>13</sup>C NMR spectrum (CDCl<sub>3</sub>): 57.32 (C(1)), 130.76 (C(3)), 123.09 (C(4)), 47.02 (C(5)), 49.56 (C(6)), 25.69 (C(7)), 27.13 (C(8)), 22.22 (C(9)), 40.98 (C(10)), 139.06, 128.13, 126.32, 127.93 (phenyl), 134.96, 129.63, 113.83, 158.37 (p-methoxyphenyl), 55.03 ppm (the CH<sub>3</sub>O group in the aryl radical at C(s). In the case of the aromatic substituents, the signals of the quaternary, ortho, meta, and para carbon atoms were given in that order. Found: C, 71.6; H, 6.9; S, 16.9%. Calculated for C22H23OS2: C, 71.7; H, 6.6; S, 17.4%.

The treatment of 20 mmole of trans, trans acetal Ia according to the method just described gives 5.9 g (87%) of hemidithioacetal IVa with  $T_m = 107-109$ °C (from a 1:3 ethanol-acetone mixture); according to the data in [8],  $T_m = 107-109$ °C. <sup>13</sup>C NMR spectrum (CDCl<sub>3</sub>): 57.20 (C(1)), 130.82 (C(3)), 122.54 (C(4)), 47.72 (C(5)), 49.23 (C(6)), 22.50 (C(7)), 26.92 (C(8)), 22.06 (C(9)), 40.78 (C(10)), 138.28, 128.26, 126.32, 127.87 (phenyl at C(3)), 142.88, 128.66, 128.04, 126.49 ppm (phenyl at C(5)).

Conversion of trans, trans-1-Methoxy-3-5-diary1-2-oxabicyclo[4.4.0]dec-3-enes (Ib, c) into 2-(4-Methoxypheny1)-4-pheny1-5,6-tetramethylene-4H-thiopyran (IIIb) and 2-Pheny1-4-

					0.3022	+0.7	979-	+ U,4	<del>1</del> 90	z = - 0	.50	Jo 					
		S <sub>(2)</sub>		C <sub>(3)</sub>			C <sub>(4)</sub>		C <sub>(5)</sub>			C <sub>(1)</sub> *		C <sub>(6)</sub> *		\$(1)*	
Plane 1 (ring A)	<u>,</u>	-0,003			0,009	-0,009		9		0,004	0,450			-0,	,339		2,267
		C <sub>(7)</sub> *			C <sub>(10)</sub> *		C <sub>(11)</sub> *		C <sub>(17)</sub> *			H <sub>(5)</sub> *		H <sub>(6)</sub> *			
		-0,154			0,059	-	-0,124			-0,898		0,949		-1,304			
		-		0.1	18x + 0	,827	y-0	.549	)z =	= 5.78	31						
	c,	:1)	C <sub>(6</sub>	,	C <sub>(7)</sub>	С	C(8) C(		0	C <sub>(10</sub>	,	s <sub>(1)</sub> *	S <sub>(2)</sub> *		C <sub>(5)</sub> *	Н	(6)*
Plane 2 (ring B)	0,2	34	-0,2	26	0,222	-0	,218	0,2	30	0,24	2	2,055	-	0,547	0,220	) - 1	1,223
		•			0.261 <i>x</i> ·	+0,9	65 <i>y</i> -	0.(	018	z = 2.76	58	•			•		
		C <sub>(11)</sub>			C <sub>(12)</sub>	2) C <sub>(13)</sub>			C <sub>(14)</sub>			C <sub>(15)</sub>		C <sub>(16)</sub>		C <sub>(</sub>	3)*
Plane 3 (ring C)		0,001			0,001 0,0		0,003		-0,006			0,005		0,002		0,	054
				0	.169 <i>x</i>	0.60	18 <i>y</i> —	0.7	56 <i>z</i>	= -12	.19	)	****				
		C <sub>(17)</sub>		(18) C <sub>(19</sub>		)	C (20)		C(21)		)	C <sub>(22)</sub>		, C <sub>(5)</sub> *			0
Plane 4 (ring D)		0,006	6 0,0	)02	02 -0,0		0,006		5 0,002		2	-0,00		09 - 0,015		2 0	,022
		1/2			1/3		1/4		2/3		2/4		T	3/4			
Angles betwe planes in de	en g	14,0			46,7	99,4			39,0		95,6			121,9			

TABLE 2. Equations of the Planar Fragments in the Molecule Ax + By + Cz = D\* and Deviations of Atoms from Them in Å 0.352x+0.797u+0.490z=-5.503

\*Atoms not involved in the calculation of the equations of the corresponding planes.

<u>methoxyphenyl)-5,6-tetramethylene-4H-thiopyran (IIIc)</u>. A suspension of 20 mmole of acetal IB in 35 ml of glacial acetic acid with an addition of 2.5 ml of acetic anhydride is saturated by a mixture of hydrogen sulfide and hydrogen chloride at 10-15°C. After 0.5 h, dissolution of original acetal Ib is observed, and after 1.5 h the reaction mixture is poured onto ice, and the precipitate formed is filtered out, washed with water, and dried in a desiccator. The precipitate is dissolved in ether and filtered, and 4H-thiopyran IIIb is precipitated from the etheral extract by ethanol. The yield is 4.6 g (69%), and  $T_m = 107-108°C$  (ethanol-ether). PMR spectrum (CDCl<sub>3</sub>): 1.65-2.20 (8H, m, methylene protons), 3.68 (3H, s, CH<sub>3</sub>O), 3.95 (1H, d, 4-H), 5.81 (1H, d, 4-H, <sup>3</sup>J<sub>3</sub>, 4 = 5.9 Hz), 6.69-7.20 ppm (9H, aromatic protons). Found: C, 79.3; H, 6.4; S, 9.6%. Calculated for C<sub>22</sub>H<sub>22</sub>OS: C, 79.0; H, 6.6; S, 9.6%.

4H-Thiopyran IIIc is obtained in analogy to the method just described from 20 mmole of acetal Ic. The yield is 4.8 g (72%), and  $T_m = 119-120^{\circ}C$  (ethanol-ether). PMR spectrum (CDCl<sub>3</sub>): 1.61-2.20(8H, m, methylene protons), 3.67 (3H, s, CH<sub>3</sub>O), 3.95 (1H, d, 4-H), 5.96 (1H, d, 3-H,  $^{3}J_{3,4} = 5.9$  Hz), 6.72-7.40 ppm (9H, m, aromatic protons. Found: C, 78.9; H, 6.4; S, 9.6%. Calculated for C<sub>22</sub>H<sub>22</sub>OS: C, 79.0; H, 6.6; S. 9.6%.

If the reaction is carried out in the absence of acetic anhydride, 0.6 g (9.5%) of 2-phenyl-4-(4-methoxyphenyl)bicyclo[3.3.1]non-2-en-9-one (VIc) with  $T_m = 174-175^\circ$  (ethanol) is recovered from the residue which does not dissolve in ether. According to [9],  $T_m = 174-175^\circ$ C. A mixed sample with a known preparation of the compound does not show melting-point depression.

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STUDY OF KINETICS AND MECHANISM OF SULFONATION OF THIOPHENE AND ITS DERIVATIVES BY COMPLEX COMPOUNDS OF SULFURIC ANHYDRIDE

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By quantitatively studying the sulfonation of thiophene and its homologs by complexes of sulfuric anhydride with ethers, amides, and trialkyl phosphates it was possible to determine kinetic and thermodynamic parameters of the process, to propose a SE2 type reaction mechanism and also to reveal a quantitative dependence of the rate constant of the sulfonation reaction of thiophene on the basicity of the complex-forming agent: The sulfonating activity of the complexes studied increases in the series - amides < trialkyl phosphates < ethers, which is the reverse of the increase in the basicity series of a donor.

Despite the large amount of quantitative information of the electrophilic substitution in the heteroaromatic ring [1, 2], no quantitative data are available in the literature\* on the sulfonation reaction of 5-membered heteroaromatic compounds, including thiophenes, which are widely used in fine organic synthesis [3], and in particular for the preparation of medicinal compounds, where thiophene sulfonic acid and its homologs are intermediate products in the synthesis of sulfamide and sulfanilamide derivatives [4, 5].

On the other hand, in recent years great attention has been paid to the study of the structure and reactivity of complex compounds of  $SO_3$ , which are of interest as mild sulfonating reagents for the sulfonation of acidophobic compounds (heterocyclic compounds, alcohols, polysaccharides, steroids, etc) [6, 7].

Therefore, besides the study of kinetics and mechanism of sulfonation of thiophene and its homologs, it was also of interest to study and quantitatively compare the reactivity of complex compounds of  $SO_3$  with different organic donors.

\*We have made a preliminary report on this problem in coauthorship with B. V. Passet in 1979 at the XVth Scientific Session of Chemistry and Technology of Organic Compounds and Sulfur-Containing Oils in Ufa.

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