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SYNTHESIS OF ISOMERIC 4- AND 5-HYDROXYLAMINOTHIAZOLIDIN-2-THIONES

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Isomeric 4- and 5-hydroxylaminothiazolidin-2-thiones were synthesized by the reaction of 1,2-aminosubstituted oximes with CS_2, and of dimeric olefin nitrosochlorides with dithiocarbamate salts. These compounds react with aldehydes and ketones to form the respective nitrones. In contrast to the 5-derivatives, the 4-hydroxylamino derivatives hydrolyze to 4-hydorxythiazolidin-2-thiones.

We have previously reported the synthesis and reactivity of the 4-hydroxylaminoimidazolidin-2-ones [1, 2]. In continuation of our studies of heterocyclic systems that contain an exocyclic hydroxylamino group we have obtained the hitherto unknown 4- and 5-hydroxylaminothiazolidin-2-thiones.

By the reaction of 1,2-aminosubstituted oximes Ia-e with CS_2 we have synthesized the respective 5-hydroxylaminothiabzlidin-2-thiones, IIa-e. The N-(2-oximinoalkyl)dithiocarbamic acids A were separated as the cyclic form B, but treatment of, e.g., IIc with alkali by analogy with the properties of the 5-hydroxythiazolidin-2-thiones [3] gave salt III of linear structure. The ^{13}C NMR spectrum of the latter in H_2O is characterized by signals at 211.0 ($C_{(1)}$=S), 161.9 ($C_{(2)}$=N), 61.8 ($C_{(3)}$), 25.8 ($C_{(4,5)}$), and 11.1 ppm ($C_{(6)}$). Neutralization of the aqueous solution of III gives the starting hydroxylamine IIc, while methylation is accompanied by formation of the linear ester IV; the ^{13}C spectrum of the latter (DMSO) contains signals at 196.4 ($C_{(1)}$=S), 158.4 ($C_{(2)}$=N), 61.5 ($C_{(3)}$), 25.5 ($C_{(4,5)}$), 17.6 (S–CH_3), and 10.0 ppm ($C_{(6)}$). The locations of the carbon signals for compounds III and IV agree with the ^{13}C NMR spectra (C=S 193.5, C=N 153.5 ppm) for the authentic linear structure, viz., N,N-dimethyl-S-(3-oximino-2-methylprop-2-yl)dithiocarbamate synthesized according to [6].

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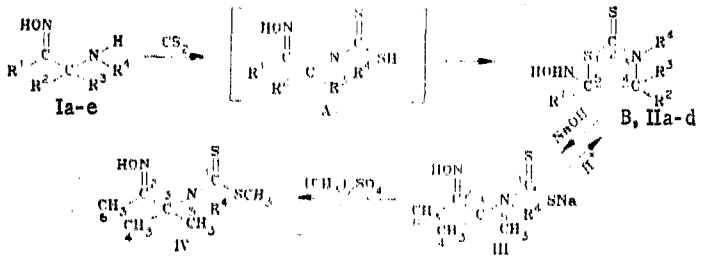
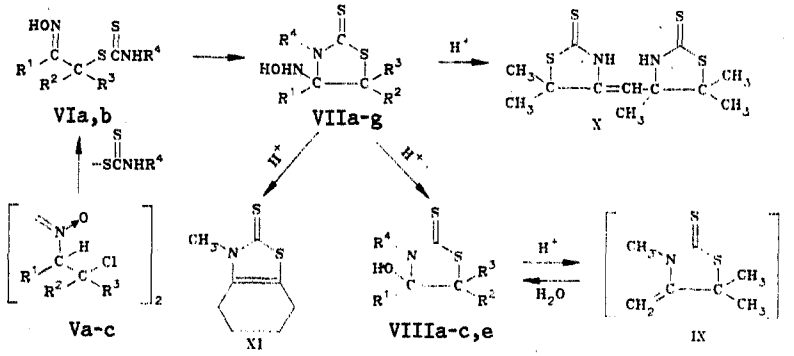


I, IIa,e $R^1 = H$, b,c $R^1 = CH_3$, d $R^1 + R^2 = (CH_2)_4$; a-c,e $R^2 = R^3 = CH_3$; a,b,d $R^4 = CH_3$, c $R^4 = H$, e $R^4 = OH$

It is of interest to note that 1,2-aminoaldoximes I with bulky substituents (R^4 = iso-C_3H_7, C_6H_{11}, $CH_2C_6H_5$) do not react with CS_2 under these conditions.

The 4-hydroxylaminothiazolidin-2-thiones VII were obtained by the reaction of the dimeric nitrosochlorides V with dithiocarbamate salts. The linear s-(2-oximinoalkyl)dithiocarbamate VI that is formed initially in this reaction was noted in the PMR spectrum ($CDCl_3$) in the reaction of potassium N-methyldithiocarbamate with 2-chloro-2-methylpropanal oxime (from the thermal conversion of nitrosochloride Va [4]). After the initial chlorooxime disappeared from the solution (as monitored by TLC), as the intensity of the signals of cyclic VIIa increased in the spectrum, viz., 1.5, 1.6 ($C(CH_3)_2$, s); 3.5 ($N{-}CH_3$, s); 4.6 ppm (CH, s), the intensities of the characteristic signals of the corresponding linear form V simultaneously decreased, viz., 1.6 ($C(CH_3)_2$, s); 3.2 ($N{-}CH_3$), d, J = 4 Hz), 7.6 ppm (N=CH, s).



V, VI a $R^1 = H$, b $R^1 = CH_3$, Vc $R^1 + R^2 = (CH_2)_4$; V, VI a,b $R^2 = R^3 = CH_3$, Vc $R^3 = H$; VI a R^4 = tert-C_4H_9, c R^4 = iso-C_3H_7; VIIa-d, VIIIa-c $R^1 = H$, VIIe,f VIIIe $R^1 = CH_3$, VIIg $R^1 + R^2 = (CH_2)_4$; VIIa–f, VIIIa-c,e $R^2 = R^3 = CH_3$, VIIg $R^3 = H$; VIIa,e,g VIIIa,e $R^4 = CH_3$, VII, VIII b $R^4 = CH_2C_6H_5$, c R^4 = iso-C_3H_7; VIIe,f $R^4 = H$

It should be noted that in [5] the products of the reaction of Va,b and the N-methyldithiocarbamate salts were mistakenly assigned a linear structure.

In contrast to the ^{13}C NMR spectra of the linear compounds III and IV, the spectra of 5- and 4-hydroxylaminothiazolidin-2-thiones II and VII (DMSO) show, instead of the C=N signals, the $C_{(5)}$ or $C_{(4)}$ signals at 87-92 ppm, along with the general C=S signals for the linear and cyclic structures (194-197 ppm for II and VII).

With increase in bulk of R^1 and R^4, e.g., in compounds VIa,b, cyclization to thiazolidin-2-thiones VII does not take place, according to the IR and NMR spectra.

In acid solution compounds Va-c,e are converted to the respective 4-hydroxythiazolidin-2-thiones VIIIa-c,e. Their IR spectra (CCl_4) show valence vibration bands at 3560 cm^{-1} (OH), and the bands at 3400 and 3590-3600 cm^{-1} (NHOH) observed for II and VII are absent. The PMR spectrum (DMSO-D_6), e.g., that of the 4-hydroxy derivative VIIIa, shows two gem-dimethyl singlets at 1.31 and 1.43, a singlet at 3.26 ($N{-}CH_3$), and two doublets (J = 7 Hz) of the CHOH protons at 5.03 and 7.05 ppm.

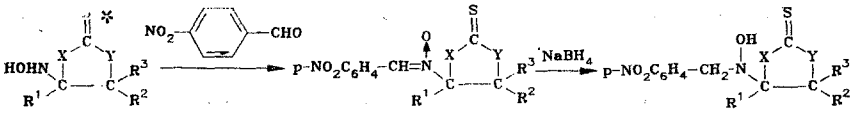
In benzene in the presence of 4-toluenesulfonic acid, 3,4,5,5-tetramethylthiazolidine VIIIe loses water reversibly to form the 4-methylene derivative IX. Thus when its solution in C_6D_6 is heated directly in the spectrometer monitor, as the intensity of the four CH_3 signals weakens (at 0.86, 0.90, 0.95, and 2.92 ppm) there appear signals at 1.08 and 2.88 ppm ($C(CH_3)_2$

and N–CH_3, respectively) and the signals of methylene protons in the form of an AB system (3.78 and 3.80 ppm, J = 3 Hz).

In concentrated acid the 4-hydroxylamine derivative, like the corresponding 4-hydroxythiazolidin-2-thione, is converted to X [3], while VIIg is converted to the 4-thiazolin-2-thione XI.

In contrast to VII, the isomeric 5-hydroxylaminothiazolidin-2-thiones II do not undergo appreciable hydrolysis in mineral acid solution. Neutralization of the hygroscopic salts yields the initial hydroxylamines II.

Condensation of isomeric II and VII with 4-nitrobenzaldehyde gives the respective nitrones XII. The presence in the UV spectrum of XII of an intense absorption band at 346 nm (log ε 4.2) and reduction to the respective N,N-disubstituted hydroxylamines XIII confirm the presence of the aryl nitrone group [7].



IIa,d VIIc XIIa-c XIIIa-c

XII, XIII a, b X=S, Y=NCH_3, c X=*iso*-C_3H_7N, Y=S; a, c R^1=H, R^2=R^3=CH_3, b R^1+R^2=$(CH_2)_4$, R^3=H

*Unclear in Russian original – Editor.

The PMR spectra of nitrones XII show the singlet of the azomethine proton in the 8.1-8.7 ppm region, whereas compounds XIII are characterized by the methylene proton signals as an AB system in the 4 ppm region (J = 15 Hz) and the OH signal at 7.1-7.9 ppm.

EXPERIMENTAL

IR spectra were recorded on a Perkin Elmer 457 instrument in KBr tablets and in CCl_4 solutions; UV spectra, on a Specord UV-vis instrument in ethanol. PMR spectra were obtained on

TABLE 1. PMR Spectra of Compounds II, IV, VI, VII

Compound	Chemical shifts, ppm (SSCC, J) (NH and OH signals, broadened)
IIa	1,31, 1,39 [6H, s, $C(CH_3)_2$], 3,08 (3H, s, NCH_3), 4,65 (1H, d, J=9 Hz, CH), 6,23 (NH, d, J=9 Hz), 7,65 (OH, s)
IIb	1,20, 1,31, 1,40 (9H, s, CH_3), 3,11 (3H, s, NCH_3), 6,18 (NH, s), 7,62 (OH, s)
IIc	1,28, 1,43 (9H, s, CH_3), 6,15 (NH, s), 7,65 (OH, s), 9,90 (NH, s)
IId	1,08—2,32 [8H, m, $(CH_2)_4$], 3,18 (3H, s, NCH_3), 4,01 (1H, m, CH), 6,03 (NH, s), 7,72 (OH, s)
IIe	1,30, 1,32 [6H, s, $C(CH_3)_2$], 4,95 (1H, d, J=6 Hz, CH), 6,31 (NH, d, J=6 Hz), 7,86 (OH, s), 10,5 (OH, s)
IV	1,62 [6H, s, $C(CH_3)_2$], 1,82 (3H, s, CH_3), 2,46 (3H, s, NCH_3), 8,73 (NH, s), 9,68 (OH, s)
VIa	1,50 [6H, s, $C(CH_3)_2$], 1,53 (9H, s, tert-C_4H_9), 7,55 (1H, s, CH=N), 8,19 (NH, s), 10,3 (OH, s)
VIb	1,20 d, 4,55 m (J=7 Hz, *iso*-C_3H_7), 1,53 [6H, s, $C(CH_3)_2$], 8,20 (NH, s), 10,42 (OH, s)
VIIa	1,53, 1,55 [6H, s, $C(CH_3)_2$], 3,38 (3H, s, NCH_3), 4,68 (1H, d, J=5 Hz, CH), 6,48 (H, d, J=5 Hz), 7,0 (OH, s)
VIIb	1,24, 1,40 [6H, s, $C(CH_3)_2$], 4,20 (1H, s, CH), 4,46, 5,62 (2H, AB system, J=15 Hz, CH_2), 6,75 (NH, s), 7,31 (5H, m, C_6H_5), 7,42 (OH, s)
VIIc	1,36, 1,52 [6H, s, $C(CH_3)_2$], 1,27 d, 1,50 d, 5,02 m (J=5 Hz, *iso*-C_3H_7), 4,52 (1H, d, J=3 Hz, CH), 6,26 (NH, s), 7,61 (OH, s)
VIId	1,38, 1,46 [6H, s, $C(CH_3)_2$], 4,23 (1H, d, J=6 Hz, CH), 6,04 (NH, d, J=6 Hz), 7,46 (OH, s), 8,93 (NH, s)
VIIe	1,40, 1,44, 1,46 (9H, s, CH_3), 3,26 (3H, s, NCH_3), 6,26 (NH, s), 7,20 (OH, s)
VIIf	1,31, 1,35, 1,43 (9H, s, CH_3), 5,96 (NH, s), 7,46 (OH, s), 9,34 (NH, s)
VIIg	1,30—2,0 [8H, m, $(CH_2)_4$], 3,04 (3H, s, NCH_3), 4,20 (1H, s, CH), 6,73 (NH, s), 7,71 (OH, s)

*Spectra of compounds IV, VIa,b, VIIIa were obtained in acetone-D_6; of VIIc, in DMSO-D_6–benzene-D_6 mixture; other compounds, in DMSO-D_6.

Varian FT-80A (80 MHz) and Bruker HX-90E (90 MHz) instruments; ^{13}C NMR spectra, on a Bruker HX-90E instrument (22.625 MHz), TMS internal standard. The course of the reactions was monitored on Silufol UV-254 sheets in 1:1 THF:hexane and 3:1 benzene:acetone systems.

The physicochemical and spectral properties of the synthesized compounds are shown in Tables 1 and 2.

The substituted aminooximes I and the dimeric nitrosochlorides V were synthesized by the procedures of [8].

5-Hydroxylaminothiazolidin-2-thiones (IIa-d). A. To a solution of 10 mmoles of aminooxime Ia-c in 4 ml of DMFA or DMSO was added 17 mmoles of CS_2. After 1 h the reaction mixture was diluted with 50 ml of water and extracted (3 × 50 ml) with ether. The extracts were dried over $MgSO_4$ and evaporated, and the residue was treated with petroleum ether to yield compounds IIa-c.

B. A mixture of 20 mmoles of methylaminooxime Id and 30 mmoles of CS_2 in 100 ml of absolute alcohol was kept for 48 h. At the end of the reaction the solvent was evaporated and the residue was recrystallized from benzene to give compound IId.

3-Hydroxy-4,4-dimethyl-5-hydroxylaminothiazolidin-2-thione (IIe). A mixture of 11 mmoles of hydroxylaminooxime Ie and 21 mmoles CS_2 in 30 ml of acetonitrile was held for 1 h at 60°. The solvent was evaporated and the residue was treated with acetone. Upon prolonged standing at 5° a precipitate formed, which was filtered off to give [N-(3-hydroxy-4,4-dimethylthiazolidin-2-thion-4-yl)-C-dimethyl] nitrone, XIV. IR spectrum (KBr): 1530, 1430, 1365, 1290, 1225, 1155, 1130, 1115, 1010, 970, 920, 815, 780 cm^{-1}. PMR spectrum (DMSO-D_6): 1.28 (3H, s, CH_3), 1.36 (3H, s, CH_3), 1.96, 2.19 (two doublets of $(CH_3)_2N$), 5.92 (1H, s, CH). The material was treated with 2 ml of conc. HCl and 1 mmole of nitrone XIV and heated to 50°. After cooling, the mixture was neutralized with dilute NaOH solution and extracted with ether. The extract was dried over $MgSO_4$ and evaporated, and the residue was treated with hexane, and filtered to yield compound IId.

S-Methyl-N-(3-oximino-2-methylbut-2-yl) dithiocarbamate (IV). To a solution of 10 mmoles of IIc and 11 mmole of NaOH in 40 ml of water was added 12 mmoles of dimethyl sulfate gradually with vigorous stirring. The precipitate was filtered off after 1 h, washed with water, and dried to give the methyl ester IV. IR spectrum (CCl_4): 3590 (OH), 3380 (NH); in KBr: 1520, 1500, 1440, 1355, 1210, 1145, 1020, 1000, 955, 945, 920, 760 cm^{-1}.

N-Tert-butyl (or isopropyl)-S-[2-methyl-3-oximinoprop-2-yl (or but-2yl)] Dithiocarbamates (VIa,b), 4-Hydroxylaminothiazolidin-2-thiones (VIIa-g). A mixture of equivalent amounts of the respective dimeric nitrosochloride V and the dithiocarbamate salt was boiled in absolute alcohol. At the end of the reaction (as monitored by TLC) the inorganic precipitate was filtered off and the solvent was evaporated. The residue was recrystallized from benzene or alcohol. IR spectrum (KBr) of VIa,b: 1520 cm^{-1} (NHC=S). ^{13}C NMR spectrum (DMSO) of VIa: 191.6 (C=S), 153.7 ppm (C=N); VIb: 191.9 (C=S), 159.3 ppm (C=N).

Hydrolysis of 4-Hydroxylaminothiazolidin-2-thiones (VIIa-c, e-g). A suspension of 10 mmoles of VII in 10 ml of conc. HCl or 50% H_2SO_4 was held at 50° for 15 min. After neutralization the reaction mixture was extracted with ether (3 × 30 ml). The extract was dried over $MgSO_4$ and evaporated, and the residue was treated with hexane to yield the respective 4-hydroxythiazolidin-2-thiones VIIIa,c,e. In the case of VIIa the product that crystallized on standing was treated with CCl_4 and filtered to yield VIIIa. In the case of VIIf the precipitate that formed in acid solution was filtered off, washed with water, and reprecipited from acetone–petroleum ether mixture to give a 46% yield of 4-(5,5-dimethyl-2-thioxo-2-thiazolidinylidenemethyl)-4,5,5-trimethylthiazolidin-2-thione X, mp 216-218° (decomposition); according to [3], mp 221° (decomposition). The IR and PMR spectra agree with those published. In the case of VIIg, the precipitate that formed in acid was filtered off, washed with water, and dried to yield 4,5-tetramethylene-3-methyl-4-thiazolidin-2-thione XI. IR spectrum (KBr): 1640 cm^{-1} (C=C). PMR spectrum (acetone-D_6): 1.81-2.51 (8H, m, $(CH_2)_4$), 3.53 ppm (3H, s, CH_3).

[N-(Thiazolidin-2-thion-5-yl)- or [N-(Thiazolidin-2-thion-4-yl)-C-(4-nitrophenyl)] Nitrones (XII). A mixture of equimolar amounts of II or VIIc with 4-nitrobenzaldehyde in benzene was boiled with or without 4-toluenesulfonic acid, respectively, until the reaction was finished (monitored by TLC). The solvent was evaporated and the residue was treated with ether to yield compound XII.

TABLE 2. Physicochemical Data for Compounds II, IV, VI-VIII, XI-XIV

Compound	mp,* °C	Found, %				Empirical formula	Calculated, %				Yield, %
		C	H	N	S		C	H	N	S	
IIa	112—113	37,4	6,3	14,4	33,4	$C_6H_{12}N_2OS_2$	37,5	6,3	14,6	33,4	84
IIb	150—151	40,6	6,7	13,6	31,0	$C_7H_{14}N_2OS_2$	40,8	6,8	13,6	31,1	30
IIc	122—124	37,1	6,5	14,6	33,7	$C_6H_{12}N_2OS_2$	37,5	6,3	14,6	33,4	45
IId	124—125	44,3	6,5	12,6	29,7	$C_8H_{14}N_2OS_2$	44,0	6,5	12,8	29,4	67
IIe	106—108	30,7	5,4	14,3	33,4	$C_6H_{10}N_2O_2S_2$	30,9	5,2	14,4	33,4	60
IV	116	41,0	6,9	13,4	33,6	$C_7H_{14}N_2OS_2$	40,8	6,8	13,6	33,4	50
VIa	104	46,5	7,6	11,9	27,3	$C_9H_{18}N_2OS_2$	46,1	7,7	12,0	27,4	50
VIb	108	46,4	7,5	12,2	27,5	$C_9H_{18}N_2OS_2$	46,1	7,7	12,0	27,4	52
VIIa	125—128	37,6	6,2	14,3	33,4	$C_6H_{12}N_2OS_2$	37,5	6,3	14,6	33,4	84
VIIb	77—79	53,7	6,3	10,3	23,9	$C_{12}H_{16}N_2OS_2$	53,7	6,0	10,4	23,9	85
VIIc	125	43,7	7,3	12,7	29,1	$C_8H_{16}N_2OS_2$	43,8	6,1	12,7	29,1	44
VIId	135—136	33,5	5,6	15,7	35,7	$C_5H_{10}N_2OS_2$	33,9	5,7	15,7	35,9	92
VIIe	118—119	40,5	7,0	13,4	31,5	$C_7H_{14}N_2OS_2$	40,7	6,8	13,6	31,1	50
VIIf	130—132	37,1	6,0	14,9	33,0	$C_6H_{12}N_2OS_2$	37,5	6,3	14,6	33,4	56
VIIg	140—141	44,3	6,7	13,0	28,9	$C_8H_{14}N_2OS_2$	44,0	6,5	12,8	29,4	50
VIIIa	34—35	40,7	6,3	8,1	36,0	$C_6H_{11}NOS_2$	40,7	6,3	7,9	36,2	40
VIIIb	126—127	56,6	6,1	5,4	25,2	$C_{12}H_{15}NOS_2$	56,9	6,0	5,5	25,3	79
VIIIc	124—125	46,7	7,0	6,6	31,2	$C_8H_{15}NOS_2$	46,8	7,4	6,8	31,2	90
VIIIe	64	44,1	7,0	7,1	33,0	$C_7H_{13}NOS_2$	43,9	7,4	7,3	33,5	70
XI	79—82	51,5	6,1	7,4	34,5	$C_8H_{11}NS_2$	51,9	6,9	7,6	34,6	56
XIIa	210	48,2	5,0	13,3	20,0	$C_{13}H_{15}N_3O_3S_2$	48,0	4,7	12,9	19,7	50
XIIb	160—162	51,4	5,0	12,1	18,5	$C_{15}H_{17}N_3O_3S_2$	51,3	4,9	12,0	18,2	60
XIIc	168	50,3	5,3	13,6	17,4	$C_{15}H_{19}N_3O_3S_2$	50,1	5,3	13,4	17,8	45
XIIIa	153—154	47,8	5,2	12,6	19,4	$C_{13}H_{17}N_3O_3S_2$	47,7	5,2	12,6	19,5	55
XIIIb	59	51,2	5,6	12,0	18,3	$C_{15}H_{19}N_3O_3S_2$	51,0	5,4	11,9	18,4	44
XIIIc	169—170	50,9	6,3	12,0	18,2	$C_{15}H_{21}N_3O_3S_2$	50,7	6,0	11,8	18,0	30
XIV	175	41,1	6,4	12,0	27,4	$C_8H_{14}N_2O_2S_2$	41,0	6,0	11,9	27,4	66

*Compounds IIe, XIIa,c and XIIIc melt with decomposition; according to [9], for VIIIa mp 57°; according to [10], for VIIIe, mp 68°.

Reduction of Nitrones XII. To a suspension of 1 mmole of XII in a 1:1 alcohol—THF mixture was added 5 mmole $NaBH_4$ in portions. The mixture was neutralized with dilute hydrochloric acid and extracted with ether (3 × 5 ml). The extracts were dried over $MgSO_4$ and evaporated and the residue was crystallized from a ether—hexane mixture to yield compound XIII.

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