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Structure of the Condensation Products of 3-Sulfanylpropionic Acid Hydrazide with Aldehydes, Ketones, and Aldoses

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Abstract—Condensation products of aliphatic aldehydes with 3-sulfanylpropionic acid hydrazide exist in solution as mixtures of linear hydrazone and cyclic 1,3,4-thiadiazepine tautomers. Hydrazones derived from 3-sulfanylpropionic acid hydrazide and aromatic aldehydes and ketones have mostly linear structures of different stereoisomers arising from *Z–E* isomerism with respect to the double C=N bond and restricted rotation about the C(O)–N bond. Condensation products of 3-sulfanylpropionohydrazide with a series of aldoses give rise to ring–chain–ring tautomeric equilibrium between α,β-isomeric pyranose structures, open-chain aldose hydrazone tautomer, and two diastereoisomeric seven-membered cyclic 1,3,4-thiadiazepine forms.

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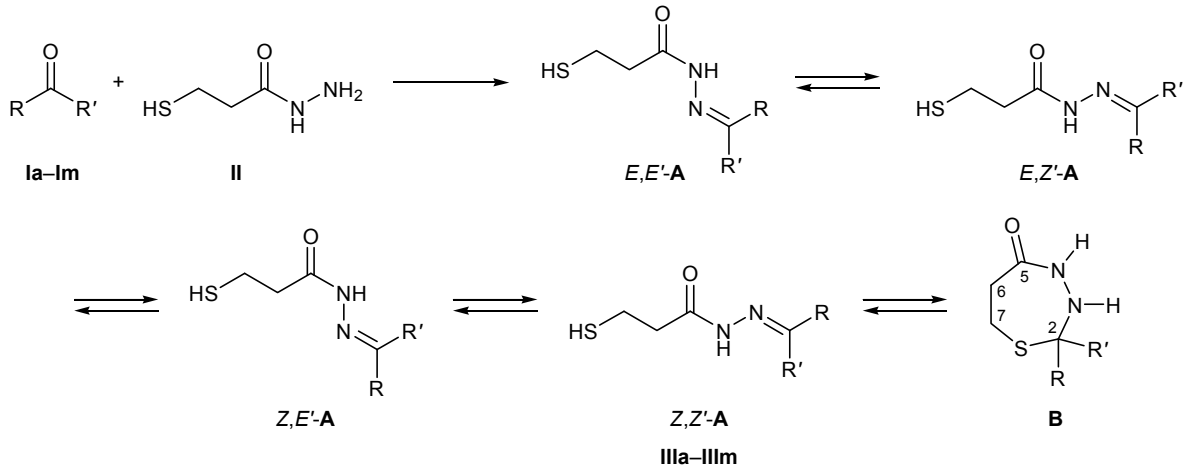
We previously showed that condensation products of carbonyl compounds with sulfanylacetic and 2-sulfanylbenzoic acid hydrazides in solution give rise to ring–chain tautomerism between linear acylhydrazone and cyclic 1,3,4-thiadiazine or 1,3,4-benzothiadiazepine structures, respectively [1–5]. In both cases, the cyclic tautomer is formed via intramolecular nucleophilic addition of the sulfanyl group at the C=N bond in the hydrazone fragment. While continuing studies in this line, we examined the ability of condensation products formed by aldehydes, ketones, and aldoses with 3-sulfanylpropionic acid hydrazide to undergo ring–chain isomerism (Schemes 1, 2).

We initially studied 3-sulfanylpropanoylhydrazones **IIIa**–**IIIj** derived from aliphatic and aromatic aldehydes and ketones. These compounds were synthesized in 40–85% yield by reaction of 3-sulfanylpropionohydrazide (**II**) with an equimolar amount of the corresponding carbonyl compounds **Ia**–**Ij** in methanol either at room temperature or on heating under reflux for a short time.

3-Sulfanylpropionylhydrazones **IIIa**–**IIIj** can exist as linear tautomer **A** and seven-membered cyclic 1,3,4-thiadiazepine structure **B**. The latter could be formed via intramolecular addition of the SH group at the C=N bond in the hydrazone fragment. Furthermore, open-chain tautomer **A** may be represented by four stereoisomers with different configurations with respect to the C=N bond (*Z–E* isomerism) and C–N bond in the amide fragment (conformational *Z′–E′* isomerism) [1, 2] (Scheme 1).

Let us consider condensation products of hydrazide **II** with acetaldehyde, propionaldehyde, and butyraldehyde (compounds **IIIa**–**IIIc**). As follows from variation of the ^{1}H NMR spectra with time, these compounds in the crystalline state have structure **A**. The ^{1}H NMR spectrum of acetaldehyde hydrazone **IIIa** in DMSO-d_6, recorded immediately after dissolution, contained two sets of signals assignable to two stereoisomers of linear tautomer **A**. The major stereoisomer (its fraction was 73% in the initial moment) was characterized by a quartet signal from the CH=N proton at δ 7.29 ppm, a triplet signal from the SH proton at 2.43 ppm, and a broadened singlet from the NH proton at δ 10.91 ppm. The corresponding signals of the minor stereoisomer (27%) were located at

Scheme 1.



R = H, R′ = Me (**a**), Et (**b**), Pr (**c**), R′ = XC_6H_4, X = 4-O_2N (**d**), 3-O_2N (**e**), 4-Br (**f**), H (**g**), 4-Me (**h**), 4-MeO (**i**), 2-HO (**j**); R = Me, R′ = Me (**k**), *i*-Pr (**l**), Ph (**m**).

δ 7.45, 2.39, and 11.02 ppm, respectively. The presence of two linear stereoisomers was also confirmed by the ^{13}C NMR data. The observed doubling of signals in the ^{1}H and ^{13}C NMR spectra of **IIIa** should be rationalized in terms of conformational *Z′–E′* isomerism of linear structure **A** due to restricted rotation about the amide C–N bond. The major isomer should be assigned *E*,*E′* configuration, and the minor one, *E*,*Z′*. We did not consider *E–Z* isomerism with respect to the hydrazone C=N bond, taking into account that acylhydrazones derived from aldehydes are characterized by preferential or exclusive *E* configuration about the C=N bond [6–10].

Signals were assigned to linear isomers *E*,*E′*-**A** and *E*,*Z′*-**A** on the basis of the known difference in the chemical shifts of the C=N and C=O carbon atoms in the ^{13}C NMR spectra of conformational isomers of acylhydrazones derived from monocarbonyl compounds: signals from the *E′* isomer are located at δ_C 145 and 170 ppm, and those of the *Z′* isomer, at δ_C 150 and 160 ppm, respectively [8]. In the ^{13}C NMR spectrum of **IIIa**, the major isomer displayed C=N and C=O signals at δ_C 144.1 and 173.3 ppm, while the corresponding signals of the minor isomer appeared at δ_C 147.9 and 167.9 ppm, respectively. Taking into account the above stated, the major isomer was assigned *E*,*E′* configuration, and the minor one, *E*,*Z′*.

After some time, signals corresponding to cyclic 1,3,4-thiadiazepine tautomer **B** appear in the ^{1}H and ^{13}C NMR spectra of compound **IIIa** in DMSO-d_6. The most characteristic signals of structure **B** (its concentration did not exceed 15%) were those from the 2-H and NH protons at δ 4.20 and 5.10 ppm, respectively, and from the sp^3-hybridized C^2 carbon atom and C^5 at δ_C 62.1 and 173.9 ppm, respectively.

Analogous patterns were observed for solutions in DMSO-d_6 of compounds **IIIb** and **IIIc** obtained by condensation of 3-sulfanylpropionic acid hydrazide with propionaldehyde and butyraldehyde. In both cases, the fraction of cyclic 1,3,4-thiadiazepine tautomer **B** did not exceed 10%, while the ratio of the *E*,*E′* and *E*,*Z′* conformers (linear structure **A**) changed insignificantly.

We can conclude that crystalline condensation products of aliphatic aldehydes with 3-sulfanylpropionohydrazide have linear structure **A** which is partially transformed into cyclic 1,3,4-thiadiazepine tautomer **B** in going to solution. In this respect, 3-sulfanylpropionylhydrazones **IIIa**–**IIIc** radically differ from the condensation products of aliphatic aldehydes with 2-sulfanylacetohydrazide, which were studied by us previously. The latter give rise to ring–chain equilibrium in solution between the linear hydrazone and six-membered cyclic 1,3,4-thiadiazine tautomers, which is strongly displaced toward the cyclic structure [5].

Let us consider now the structure of condensation products of 3-mercaptopropionic acid hydrazide (**II**) with aromatic aldehydes (compounds **IIId**–**IIIj**). The ^{1}H NMR spectrum of *N′*-(4-nitrobenzylidene)-3-sulfanylpropionohydrazide (**IIId**) in DMSO-d_6, recorded immediately after dissolution, contained signals assignable to seven-membered 1,3,4-thiadiazepine tautomer **B**. Protons in the methylene groups of structure **B** resonated at δ 2.32 and 2.70 ppm, the 2-H signal

appeared at δ 5.38 ppm, and two NH proton signals (broadened singlets) were located at δ 4.21 and 9.02 ppm. In the ^{13}C NMR spectrum we observed a signal at δ_C 76.6 ppm due to the sp^3-hybridized C^2 atom. After several hours, signals belonging to linear hydrazone tautomer **A** appeared in the ^1H NMR spectrum. These signals were doubled, indicating the existence of several stereoisomers. The major stereoisomer of linear tautomer **A** (26%) was characterized by signals at δ 8.08 (CH=N), 2.74 and 3.00 (CH_2), 2.43 (t, SH), and 11.65 ppm (br.s, NH). The CH=N and NH protons of the minor stereoisomer resonated at δ 8.24 and 11.72 ppm, respectively. Insofar as the intensity of signals belonging to 1,3,4-thiadiazepine tautomer **B** in the ^1H NMR spectrum of **IIId**, recorded immediately after dissolution, was considerably higher than the intensity of the same signals in the spectra recorded several hours later, we presumed that compound **IIId** in the crystalline state has cyclic 1,3,4-thiadiazepine structure **B**.

In 7 days after dissolution in DMSO-d_6, the ^1H NMR spectrum of **IIId** no longer changed, and it contained only signals corresponding to linear structure **A** with a stereoisomer ratio of 67:23. Taking into account published data [6, 7], according to which acylhydrazones derived from aromatic aldehydes exist preferentially or exclusively as *E* isomers with respect to the C=N bond, the observed two sets of signals in the ^1H NMR spectrum of the linear tautomer **A** of compound **IIId** should be assigned to different conformers (*E*′–*Z*′ isomerism) arising from restricted rotation about the amide C–N bond. Here, the major conformer has *E*,*E*′ configuration, and it can be isolated as individual crystalline substance by removal of the solvent. Thus we revealed gradual irreversible transformation of cyclic 1,3,4-thiadiazepine tautomer **B** of compound **IIId** into linear *N*′-(3-sulfanylpropionyl) hydrazone **A**.

Isomeric composition of compound **IIIg** in different solvents in 48 h after dissolution

Solvent	Isomeric composition of open-chain structure **A**, %		
	E,*E*′	*E*,*Z*′	*Z*,*E*′
Chloroform-*d*	78	19	3
Pyridine-d_5	70	26	4
Acetonitrile-d_3	65	31	4
DMF-d_7	63	32	5
DMSO-d_6	61	32	7

No cyclic 1,3,4-thiadiazepine structure **B** was detected in DMSO-d_6 for 3-sulfanylpropionylhydrazones **IIIe**–**IIIj** derived from other aromatic aldehydes. Crystalline compounds **IIIe**–**IIIj** had structure *E*,*E*′-**A**, while in solution equilibrium mixtures of three linear stereoisomers **A** were formed. The *E*–*Z* equilibrium (with respect to the C=N bond) was strongly displaced toward the *E* isomer for all compounds **IIIb**–**IIIj**: the fraction of the corresponding *Z* isomer did not exceed 10%. The ratio between the *E*′ and *Z*′ conformers (with respect to the C–N bond) of the *E* isomer was almost independent on the nature of substituent in the aromatic ring. Obviously, the *E*,*E*′ and *E*,*Z*′ structures are characterized by identical conjugation systems which are similarly affected by electronic properties of the substituent in the aromatic ring.

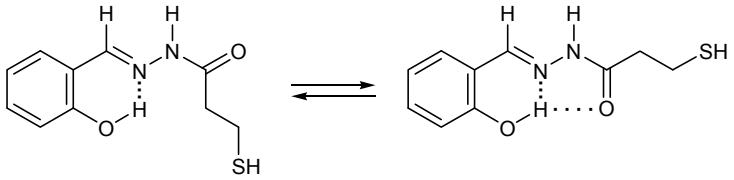
Using as example compound **IIIg** (condensation product of 3-sulfanylpropionohydrazide with benzaldehyde) we examined the relation between the isomer composition and solvent nature (see table). In all cases, structure *Z*,*E*′-**A** was detected. Its fraction increased in going to strongly basic polar solvents like DMSO-d_6 and DMF-d_7, but it did not exceed 10%. Replacement of weakly polar $CDCl_3$ by polar solvents favored formation of the *E*,*Z*′-**A** stereoisomer.

The conformational composition of linear tautomer **A** may change to an appreciable extent upon introduction into the aromatic ring of the aldehyde component of substituents capable of being involved in intramolecular hydrogen bond. For example, the *E*,*Z*′ structure predominates in solution for 3-sulfanylpropionylhydrazone **IIIj** obtained from salicylaldehyde. Presumably, this is the result of stabilization of the *E*,*Z*′ conformer due to formation of additional intramolecular hydrogen bond between the OH and C=O group. It should be noted that hydrogen bonding between the OH and C=N groups is possible in both conformers with respect to the amide C–N bond.

Thus the condensation products of aromatic aldehydes with 3-sulfanylpropionic acid hydrazide in the crystalline state have linear hydrazone structure represented by isomer *E*,*E*′-**A**, whereas in solution they give rise to equilibrium mixtures of three linear stereoisomers. Increase in the solvent polarity favors formation of the *Z*′ conformer, indicating that it is more polar than the corresponding *E*′ conformer.

It was interesting to compare tautomeric behaviors of 3-sulfanylpropionylhydrazones **IIId**–**IIIj** and condensation products of aromatic aldehydes with 2-sulfanylacetic acid hydrazide, studied previously [5]. The

Scheme 2.



E,*E*′-**A** **IIIj** *E*,*Z*′-**A**

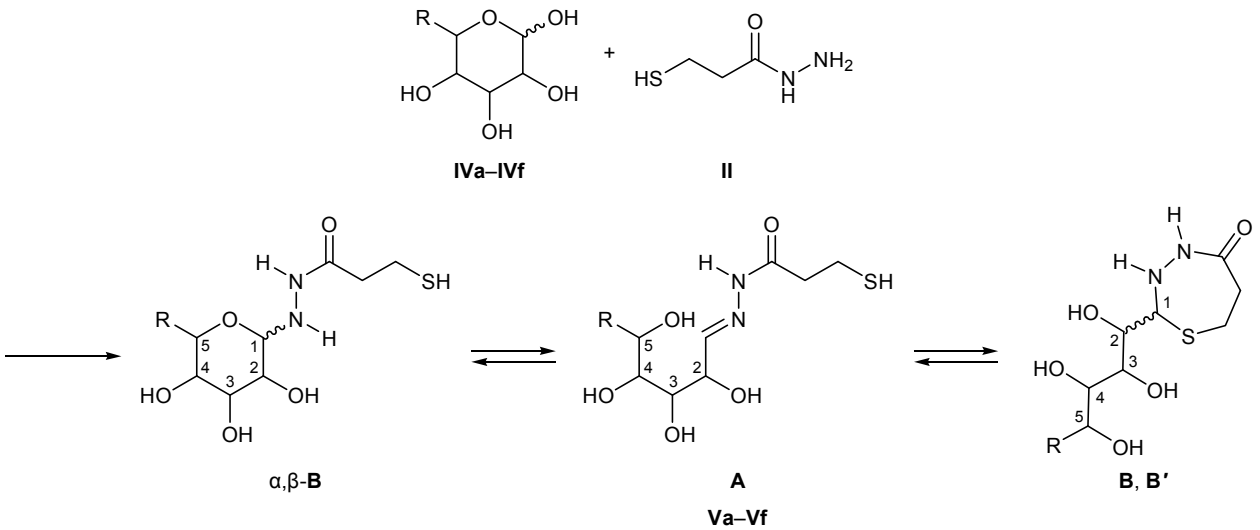
latter give rise to ring–chain tautomerism between the linear hydrazone and six-membered cyclic 1,3,4-thiadiazine structures. In contrast, even traces of seven-membered 1,3,4-thiadiazepine tautomer **B** were detected for hydrazones **IIId**–**IIIj**. In both cases, linear tautomers **A** are stabilized due to participation of the aromatic ring in the π–*p*–π conjugation system with the acylhydrazone fragment [5–8].

Compounds **IIIk**–**IIIm** were obtained by condensation of 3-sulfanylpropionic acid hydrazide with ketones **Ik**–**Im**. In the ^{1}H NMR spectrum of acetone hydrazone **IIIk** in $CDCl_3$ we observed two sets of signals corresponding to two open-chain hydrazone conformers *E*′ and *Z*′, the former prevailing (87%). The *E*′ conformer displayed a downfield signal at δ 8.83 ppm from the NH proton, two multiplet signals at δ 2.97 and 2.81 ppm from the C**H**$_2$SH and CH_2CO methylene protons, respectively, two singlets at δ 1.84 and 1.97 ppm due to methyl protons, and a triplet at δ 1.67 ppm from the SH proton. Only the signals at δ 2.07 and 1.90 ppm (CH_3) can be reliably assigned to the minor *Z*′ isomer (13%). As might be expected, the fraction of more polar *Z*′ isomer increases in going to strongly polar DMSO-d_6.

Increase in the size of the terminal substituent in going from acetone hydrazone **IIIk** to 3-methylbutan-2-one and acetophenone derivatives **IIIl** and **IIIm** (isopropyl and phenyl group, respectively) displaces the conformational equilibrium toward the *E*,*E*′ stereoisomer. Like condensation products of 3-sulfanylpropionic acid hydrazide with aromatic aldehydes, no cyclic 1,3,4-thiadiazepine tautomer **B** was detected for ketone acylhydrazones **IIIk**–**IIIm**. This feature strongly differentiates compounds **IIIk**–**IIIm** from previously studied condensation products of alkyl methyl ketones with sulfanylacetic acid hydrazide [2], which were shown to exist in solution as equilibrium mixtures of linear sulfanylacetylhydrazone and six-membered cyclic 1,3,4-thiadiazine tautomers.

It seems that certain spatial accessibility of the C=N bond for intramolecular nucleophilic attack by the SH group is a necessary condition for the formation of cyclic 1,3,4-thiadiazepine tautomer **B** of 3-sulfanylpropionylhydrazones. Such condition may be achieved with condensation products of 3-sulfanylpropionic acid hydrazide with aliphatic aldehydes or aldoses which can be regarded as latent aliphatic aldehydes. In fact, our assumptions were completely confirmed by study-

Scheme 3.



IVa–IVf **II**

α,β-**B** **A** **B**, **B′**

Va–Vf

R = H, L-arabinose (**a**), D-xylose (**b**), D-ribose (**c**); R = CH_2OH, D-galactose (**d**), D-glucose (**e**), D-mannose (**f**).

ing the structure of 3-sulfanylpropionylhydrazones derived from some aldoses (Scheme 3). Hydrazones **Va**–**Vf** were obtained by condensation of hydrazide **II** with L-arabinose (**IVa**), D-xylose (**IVb**), D-ribose (**IVc**), D-galactose (**IVd**), D-glucose (**IVe**), and D-mannose (**IVf**). Compounds **Va**–**Vf** were expected to exhibit complicated tautomeric behavior due to their ability to undergo cyclization with formation of both six-membered pyranose structure **C** and seven-membered 1,3,4-thiadiazepine structure **B**. It should also be taken into account that both cyclic and linear tautomers can exist as two stereoisomers (α- and β-anomers for structure **C**, *Z*′ and *E*′ conformers for structure **A**, and 2*R*- and 2*S*-disatereoisomers of **B**).

Different forms of compounds **Va**–**Vf** were identified by ^{13}C NMR spectroscopy. Pyranose structure **A** might be expected to display a signal from the anomeric carbon atom (C^1) at δ_C 85–90 ppm. The corresponding signal of seven-membered thiadiazepine tautomer **B** should be located in a stronger field, at δ_C 65–70 ppm, which is typical of an sp^3-hybridized carbon atom connected to sulfur and nitrogen in a seven-membered ring [4]. Finally, linear structure **A** should give rise to a signal at δ_C 150 ppm from the C=N carbon atom.

The NMR spectra of solutions of 3-sulfanylpropionylhydrazones **Va**–**Vf** in D_2O, recorded immediately after dissolution, contained signals assignable to pyranose structure **A**. Therefore, we presumed that compounds **Va**–**Vf** in the crystalline state have analogous structure. After 48 h, i.e., when the spectral patterns of solutions of **Va**–**Vf** in D_2O no longer changed (equilibrium was attained), signals corresponding to both cyclic tautomers **B** and **C** and linear hydrazone structure were present, and each cyclic tautomer was a mixture of two stereoisomers. We failed to determine configuration of the C^2 atom (*R* or *S*) in thiadiazepine structure **B**.

Analysis of the ^{13}C NMR spectra allowed us to propose additional parameters for identification of cyclic tautomers **B** and **C**. Signal from the CH_2S carbon atom in the pyran ring of tautomer **C** is located in a stronger field relative to the corresponding signal of the thiadiazepine tautomer **B**. Signals from carbon atoms in the CH_2CO group of tautomers **B** and **C** show the reverse relation.

Thus the condensation products of 3-sulfanylpropionic acid hydrazide with aldoses have pyranose structure **C** in the crystalline state. Upon dissolution in D_2O, they are partially converted into linear hydrazone tautomer **A** and cyclic 1,3,4-thiadiazepine tautomer **B**, giving rise to complex ring–chain–ring tautomeric equilibrium. The state of the equilibrium varies over a wide range: from **C**:**A**:**B** ratio 88:2:10 for glucose derivative **Ve** to 52:8:40 for xylose derivative **Va**.

The ability to undergo ring–chain–ring tautomeric transformation involving two different cyclic structures makes 3-sulfanylpropionylhydrazones **Va**–**Vf** similar to the condensation products of aldoses with sulfanylacetic and benzothioic acid hydrazides [11, 12]. The latter were found to strongly tend to form cyclic 1,3,4-thiadiazine and dihydro-1,3,4-thiadiazole structures, respectively, via intramolecular nucleophilic addition of the SH group at the hydrazone C=N bond.

The results of the present study may be interesting from the practical viewpoint, e.g., for the design of new radioprotective agents and complexones for colloidal noble metals, as well as of polymeric materials for technics, medicine, and biology [13–15].

EXPERIMENTAL

The ^1H and ^{13}C NMR spectra were recorded on a Bruker AV-400 and AT-500 spectrometers at 400 and 125 MHz, respectively, using hexamethyldisiloxane as internal reference. The compositions of tautomeric mixtures were determined from the intensity ratios of the corresponding signals in the ^1H NMR spectra. The progress of reactions and the purity of products were monitored by TLC on Silufol UV-254 plates using benzene–acetone (4:1; **IIIa**–**IIIm**) or ethyl acetate–pyridine–water (12:5:4; **Va**–**Ve**) as eluent. 3-Sulfanylpropionic acid hydrazide (**II**) was synthesized according to the procedure described in [16].

Aldehyde and ketone 3-sulfanylpropionylhydrazones IIIa–IIIm. A mixture of 15 mmol of carbonyl compound **Ia**–**Im** and 1.20 g (10 mmol) of 3-sulfanylpropionic acid hydrazide (**II**) in 50 ml of methanol was kept for 2 h at 25°C. The precipitate was filtered off, washed with diethyl ether, dried, and recrystallized from benzene–petroleum (1:4).

***N*′-Ethylidene-3-sulfanylpropionohydrazide (IIIa).** Yield 55%, mp 84–87°C. ^1H NMR spectrum (DMSO-d_6), δ, ppm: *E*,*E*′-**A** (62%): 1.85 d (CH_3, J = 5.3 Hz), 2.43 t (SH, J = 6.5 Hz), 2.78 m (CH_2CO), 2.91 m (CH_2SH), 7.29 q (HC=N, J = 5.3 Hz), 10.91 br.s (NH); *E*,*Z*′-**A** (23%): 1.83 d (CH_3, J = 5.5 Hz), 2.39 t (SH, J = 6.7 Hz), 2.66 m (CH_2CO), 2.94 m (CH_2SH), 7.45 q (HC=N, J = 5.5 Hz), 11.02 br.s (NH); **B** (15%): 1.38 d (CH_3, J = 6.3 Hz),

2.20 m (6-H), 2.32 m (7-H), 4.19 q (2-H, J = 6.3 Hz), 5.09 br.s (NH), 9.34 br.s (NH). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: *E*,*E*′-**A**: 18.1 (CH_2S), 19.3 (CH_3), 36.6 (**C**H_2CO), 144.1 (C=N), 173.3 (C=O); *E*,*Z*′-**A**: 18.3 (CH_2S), 19.2 (CH_3), 36.7 (**C**H_2CO), 147.9 (C=N), 167.0 (C=O); **B**: 20.8 (CH_3), 23.7 (C^7), 33.1 (C^6), 62.1 (C^2), 173.9 (C^5). Found, %: C 40.90; H 6.96; N 19.08. $C_5H_{10}N_2OS$. Calculated, %: C 41.07; H 6.89; N 19.16.

***N*′-Propylidene-3-sulfanylpropionohydrazide (IIIb).** Yield 60%, mp 53–56°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: *E*,*E*′-**A** (66%): 1.00 t (CH_3, J = 7.5 Hz), 2.34 m (CH_2), 2.40 t (SH, J = 6.4 Hz), 2.67 m (CH_2CO), 2.88 m (C**H**$_2$SH), 7.31 t (HC=N, J = 4.5 Hz), 10.94 br.s (NH); *E*,*Z*′-**A** (25%): 0.98 t (CH_3, J = 7.5 Hz), 2.34 m (CH_2), 2.35 t (SH, J = 6.6 Hz), 2.75 m (CH_2CO), 2.91 m (C**H**$_2$SH), 7.46 t (HC=N, J = 4.7 Hz), 11.01 br.s (NH); **B** (9%): 0.93 t (CH_3, J = 7.5 Hz), 2.23 m (6-H), 2.27 m (CH_2), 2.35 m (7-H), 4.15 t (2-H, J = 6.3 Hz), 5.27 br.s (NH), 9.34 br.s (NH). Found, %: C 48.32; H 8.03; N 15.97. $C_7H_{14}N_2OS$. Calculated, %: C 48.25; H 8.10; N 16.08.

***N*′-Butylidene-3-sulfanylpropionohydrazide (IIIc).** Yield 70%, viscous oily substance. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: *E*,*E*′-**A** (68%): 0.95 t (CH_3, J = 7.5 Hz), 1.53 m (CH_2), 2.23 m (CH_2), 2.42 t (SH, J = 6.5 Hz), 2.74 m (CH_2CO), 2.89 m (C**H**$_2$SH), 7.28 t (HC=N, J = 4.5 Hz), 10.93 br.s (NH); *E*,*Z*′-**A** (24%): 0.92 t (CH_3, J = 7.5 Hz), 1.53 m (CH_2), 2.19 m (CH_2), 2.34 t (SH, J = 6.6 Hz), 2.68 m (CH_2CO), 2.93 m (C**H**$_2$SH), 7.43 t (HC=N, J = 4.7 Hz), 11.00 br.s (NH); **B** (8%): 0.88 t (CH_3, J = 7.5 Hz), 1.34 m (CH_2), 2.17 m (CH_2), 2.24 m (6-H), 2.34 m (7-H), 4.23 t (2-H, J = 6.0 Hz), 5.25 br.s (NH), 9.36 br.s (NH). Found, %: C 45.06; H 7.47; N 17.54. $C_6H_{12}N_2OS$. Calculated, %: C 44.97; H 7.55; N 17.48.

***N*′-(4-Nitrobenzylidene)-3-sulfanylpropionohydrazide (IIId, A).** Yield 95%, mp 209–213°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: *E*,*E*′-**A** (75%): 2.43 t (SH, J = 8.1 Hz), 2.74 m (CH_2CO), 3.00 m (C**H**$_2$SH), 7.68–8.18 m (H_{arom}), 8.08 s (HC=N), 11.65 br.s (NH); *E*,*Z*′-**A** (25%): 2.32 t (SH, J = 8.3 Hz), 2.65 m (CH_2CO), 3.02 m (C**H**$_2$SH), 8.24 s (HC=N), 11.72 br.s (NH). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: *E*,*E*′-**A**: 19.0 (CH_2S), 36.4 (**C**H_2CO), 123.4–140.6 (H_{arom}), 143.8 (C=N), 173.0 (C=O); *E*,*Z*′-**A**: 19.6 (CH_2S), 38.4 (**C**H_2CO), 147.9 (C=N), 167.4 (C=O). Found, %: C 47.47; H 4.33; N 16.63. $C_{10}H_{11}N_3O_3S$. Calculated, %: C 47.42; H 4.38; N 16.59.

2-(4-Nitrophenyl)hexahydro-1,3,4-thiadiazepin-5-one (IIId, B). Yield 70%, mp 110–111°C. ^{1}H NMR spectrum (DMSO-d_6; immediately after dissolution), δ, ppm: 2.32 m (6-H), 2.70 m (7-H), 4.21 s (2-H), 5.38 br.s (NH), 7.68–8.18 m (H_{arom}), 9.02 br.s (NH). ^{13}C NMR spectrum (DMSO-d_6; immediately after dissolution), δ_C, ppm: 25.6 (C^7), 34.1 (C^6), 76.6 (C^2), 170.1 (C^5). Found, %: C 47.39; H 4.43; N 16.50. $C_{10}H_{11}N_3O_3S$. Calculated, %: C 47.42; H 4.38; N 16.59.

***N*′-(3-Nitrobenzylidene)-3-sulfanylpropionohydrazide (IIIe).** Yield 80%, mp 209–110°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: *E*,*E*′-**A** (65%): 2.44 t (SH, J = 7.5 Hz), 2.75 m (CH_2CO), 3.00 m (C**H**$_2$SH), 7.73–8.10 m (H_{arom}), 8.49 s (HC=N), 11.60 br.s (NH); *E*,*Z*′-**A** (28%): 2.41 t (SH, J = 8.3 Hz), 2.56 m (CH_2CO), 3.02 m (C**H**$_2$SH), 8.50 s (HC=N), 11.69 br.s (NH); *Z*,*E*′-**A** (7%): 2.67 m (CH_2CO), 3.09 m (C**H**$_2$SH), 8.41 s (HC=N), 11.63 br.s (NH). Found, %: C 47.47; H 4.33; N 16.67. $C_{10}H_{11}N_3O_3S$. Calculated, %: C 47.42; H 4.38; N 16.59.

***N*′-(4-Bromobenzylidene)-3-sulfanylpropionohydrazide (IIIf).** Yield 85%, mp 179–189°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: *E*,*E*′-**A** (62%): 2.33 t (SH, J = 8.1 Hz), 2.73 m (CH_2CO), 2.95 m (C**H**$_2$SH), 7.74–8.11 m (H_{arom}), 7.94 s (HC=N), 11.40 br.s (NH); *E*,*Z*′-**A** (35%): 2.30 t (SH, J = 8.4 Hz), 2.52 m (CH_2CO), 3.00 m (C**H**$_2$SH), 8.13 s (HC=N), 11.46 br.s (NH); *Z*,*E*′-**A** (3%): 2.63 m (CH_2CO), 3.05 m (C**H**$_2$SH), 7.87 s (HC=N), 11.43 br.s (NH). Found, %: C 41.78; H 3.91; N 9.72. $C_{10}H_{11}BrN_2OS$. Calculated, %: C 41.82; H 3.86; N 9.75.

***N*′-Benzylidene-3-sulfanylpropionohydrazide (IIIg).** Yield 65%, mp 107–110°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: *E*,*E*′-**A** (61%): 2.41 t (SH, J = 7.8 Hz), 2.75 m (CH_2CO), 2.96 m (C**H**$_2$SH), 7.70–8.13 m (H_{arom}), 7.99 s (HC=N), 11.38 br.s (NH); *E*,*Z*′-**A** (32%): 2.39 t (SH, J = 8.1 Hz), 2.53 m (CH_2CO), 3.02 m (C**H**$_2$SH), 8.17 s (HC=N), 11.45 br.s (NH); *Z*,*E*′-**A** (7%): 2.65 m (CH_2CO), 3.06 m (C**H**$_2$SH), 7.93 s (HC=N), 11.41 br.s (NH). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: *E*,*E*′-**A**: 19.1 (CH_2S), 36.5 (**C**H_2CO), 126.8–134.3 (C_{arom}), 143.0 (C=N), 172.6 (C=O); *E*,*Z*′-**A**: 19.8 (CH_2S), 38.4 (**C**H_2CO), 146.3 (C=N), 167.0 (C=O); *Z*,*E*′-**A**: 19.8 (CH_2S), 38.5 (**C**H_2CO), 143.2 (C=N), 172.3 (C=O). Found, %: C 57.72; H 5.77; N 13.41. $C_{10}H_{12}N_2OS$. Calculated, %: C 57.67; H 5.81; N 13.45.

***N*′-(4-Methylbenzylidene)-3-sulfanylpropionohydrazide (IIIh).** Yield 75%, mp 138–141°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: *E*,*E*′-**A** (62%): 2.32 s (CH_3), 2.45 t (SH, J = 7.7 Hz), 2.76 m (CH_2CO), 2.96 m (C**H**$_2$SH), 7.58–7.64 m (H_{arom}), 7.98 s (HC=N),

11.32 br.s (NH); *E*,*Z*′-**A** (35%): 2.30 s (CH_3), 2.41 t (SH, *J* = 8.0 Hz), 2.54 m (CH_2CO), 3.05 m (C**H**$_2$SH), 8.15 s (HC=N), 11.39 br.s (NH); *Z*,*E*′-**A** (3%): 2.31 s (CH_3), 2.66 m (CH_2CO), 3.06 m (C**H**$_2$SH), 7.92 s (HC=N), 11.36 br.s (NH). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: *E*,*E*′-**A**: 18.8 (CH_2S), 20.7 (CH_3), 36.2 (**C**H_2CO), 126.4–131.5 (H_{arom}), 142.9 (C=N), 172.4 (C=O); *E*,*Z*′-**A**: 19.4 (CH_2S), 20.7 (CH_3), 38.1 (**C**H_2CO), 146.3 (C=N), 167.6 (C=O); *Z*,*E*′-**A**: 19.4 (CH_2S), 38.2 (**C**H_2CO), 143.8 (C=N), 172.1 (C=O). Found, %: C 59.38; H 6.39; N 12.56. $C_{11}H_{14}N_2OS$. Calculated, %: C 59.43; H 6.35; N 12.60.

***N*′-(4-Methoxybenzylidene)-3-sulfanylpropionohydrazide (IIIi).** Yield 70%, mp 127–130°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: *E*,*E*′-**A** (61%): 2.41 t (SH, *J* = 8.0 Hz), 2.73 m (CH_2CO), 2.93 m (C**H**$_2$SH), 3.78 s (CH_3O), 6.98–7.60 m (H_{arom}), 7.93 s (HC=N), 11.23 br.s (NH); *E*,*Z*′-**A** (37%): 2.39 t (SH, *J* = 8.3 Hz), 2.52 m (CH_2CO), 3.02 m (C**H**$_2$SH), 3.82 s (CH_3O), 8.10 s (HC=N), 11.30 br.s (NH); *Z*,*E*′-**A** (2%): 2.62 m (CH_2CO), 3.04 m (C**H**$_2$SH), 3.76 s (CH_3O), 7.87 s (HC=N), 11.26 br.s (NH). Found, %: C 55.49; H 5.87; N 11.80. $C_{11}H_{14}N_2O_2S$. Calculated, %: C 55.44; H 5.92; N 11.76.

***N*′-(2-Hydroxybenzylidene)-3-sulfanylpropionohydrazide (IIIj).** Yield 65%, mp 152–154°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: *E*,*E*′-**A** (37%): 2.42 t (SH, *J* = 7.9 Hz), 2.74 m (CH_2CO), 2.93 m (C**H**$_2$SH), 6.85–7.63 m (H_{arom}), 8.28 s (HC=N), 9.00 br.s (OH), 11.33 br.s (NH); *E*,*Z*′-**A** (62%): 2.40 t (SH, *J* = 8.0 Hz), 2.55 m (CH_2CO), 3.04 m (C**H**$_2$SH), 8.36 s (HC=N), 11.39 br.s (NH), 11.67 br.s (OH); *Z*,*E*′-**A** (1%): 8.15 s (HC=N), 11.12 br.s (NH). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: *E*,*E*′-**A**: 19.0 (CH_2S), 36.5 (**C**H_2CO), 116.2–157.4 (C_{arom}), 142.2 (C=N), 172.3 (C=O); *E*,*Z*′-**A**: 19.6 (CH_2S), 38.2 (**C**H_2CO), 146.9 (C=N), 166.8 (C=O). Found, %: C 53.50; H 6.43; N 12.54. $C_{10}H_{12}N_2O_2S$. Calculated, %: C 53.55; H 5.39; N 12.49.

***N*′-Isopropylidene-3-sulfanylpropionohydrazide (IIIk).** Yield 60%, mp 94–96°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: *E*′-**A** (55%): 1.82 s (CH_3), 1.89 s (CH_3), 2.31 t (SH, *J* = 7.5 Hz), 2.67 m (CH_2CO), 2.92 m (C**H**$_2$SH), 10.09 br.s (NH); *Z*′-**A** (45%): 1.83 s (CH_3), 1.90 s (CH_3), 2.26 t (SH, *J* = 7.8 Hz), 2.60 m (CH_2CO), 2.95 m (C**H**$_2$SH), 10.01 br.s (NH). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: *E*′-**A**: 17.1 (CH_3), 19.1 (CH_2S), 25.2 (CH_3), 36.7 (**C**H_2CO), 150.3 (C=N), 172.6 (C=O); *Z*′-**A**: 17.6 (CH_3), 20.0 (CH_2S), 25.0 (CH_3), 38.0 (**C**H_2CO), 155.1 (C=N), 166.8 (C=O). Found, %: C 45.03; H 7.50; N 7.50. $C_6H_{12}N_2OS$. Calculated, %: C 44.97; H 7.55; N 17.48.

***N*′-(3-Methylbutan-2-ylidene)-3-sulfanylpropionohydrazide (IIIl).** Yield 70%, mp 69–71°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: *E*,*E*′-**A** (78%): 1.01 d (6H, CH_3, *J* = 6.9 Hz) 1.79 s (CH_3), 2.33 t (SH, *J* = 8.0 Hz), 2.43 m (SH), 2.68 m (CH_2CO), 2.81 m (C**H**$_2$SH), 10.05 br.s (NH); *E*,*Z*′-**A** (19%): 1.00 d (6H, CH_3, *J* = 7.0 Hz), 1.77 s (CH_3), 2.31 t (SH, *J* = 8.3 Hz), 2.43 m (SH), 2.52 m (CH_2CO), 2.92 m (C**H**$_2$SH), 9.93 br.s (NH); *Z*,*E*′-**A** (3%): 1.79 s (CH_3), 2.61 m (CH_2CO), 2.95 m (C**H**$_2$SH), 9.98 br.s (NH). Found, %: C 50.98; H 8.51; N 14.92. $C_8H_{16}N_2OS$. Calculated, %: C 51.03; H 8.56; N 14.88.

***N*′-(1-Phenylethylidene)-3-sulfanylpropionohydrazide (IIIm).** Yield 65%, mp 114–116°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: *E*,*E*′-**A** (62%): 2.23 s (CH_3), 2.40 t (SH, *J* = 7.7 Hz), 2.75 m (CH_2CO), 2.99 m (C**H**$_2$SH), 7.39–7.76 m (H_{arom}), 10.56 br.s (NH); *E*,*Z*′-**A** (33%): 2.26 s (CH_3), 2.33 t (SH, *J* = 8.0 Hz), 2.66 m (CH_2CO), 3.08 m (C**H**$_2$SH), 10.40 br.s (NH); *Z*,*E*′-**A** (5%): 2.21 s (CH_3), 2.71 m (CH_2CO), 3.08 m (C**H**$_2$SH), 10.53 br.s (NH). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: *E*,*E*′-**A**: 13.5 (CH_3), 19.1 (CH_2S), 37.0 (**C**H_2CO), 123.7–131.8 (C_{arom}), 147.2 (C=N), 173.4 (C=O); *E*,*Z*′-**A**: 14.2 (CH_3), 20.0 (CH_2S), 37.5 (**C**H_2CO), 151.1 (C=N), 167.3 (C=O); *Z*,*E*′-**A**: 13.3 (CH_3), 20.0 (CH_2S), 38.2 (**C**H_2CO), 147.4 (C=N), 170.3 (C=O). Found, %: C 59.37; H 6.39; N 12.57. $C_{11}H_{14}N_2OS$. Calculated, %: C 59.43; H 6.35; N 12.60.

Aldose 3-sulfanylpropionylhydrazones Va–Ve (*general procedure*). The corresponding carbohydrate **IV**, 10 mmol, was added to a solution of 1.20 g (10 mmol) of 3-sulfanylpropionic acid hydrazide (**II**) in 25 ml of methanol, and the mixture was heated for 1 h under reflux. The solvent was removed under reduced pressure, the residue was washed with diethyl ether, and the crystalline material was filtered off, dried under reduced pressure, and stored in a desiccator over P_2O_5.

L-Arabinose 3-sulfanylpropionylhydrazone (Va). Yield 55%, mp 146–148°C. ^{13}C NMR spectrum (D_2O), δ_C, ppm: **A** (7%): 152.0 (C=N), 169.8 (C=O); **B**, **B**′ (30%): 25.0 and 25.5 (CH_2S), 33.4 and 34.2 (**C**H_2CO), 63.7 (C^5), 67.5 (C^1), 69.6 (C^4), 70.2 (C^3), 70.5 (C^2), 172.1 (C=O); α-**C** (10%): 19.3 (CH_2SH), 36.8 (**C**H_2CO), 61.8 (C^5), 68.2 (C^2), 69.1 (C^3), 70.0 (C^4), 86.0 (C^1), 172.6 (C=O); β-**C** (53%): 19.3 (CH_2S), 37.0 (**C**H_2CO), 62.5 (C^5), 68.8 (C^4), 70.3 (C^2), 72.0 (C^3), 89.8 (C^1), 172.6 (C=O). Found, %: C 38.03; H 6.44;

N 11.06. $C_8H_{16}N_2O_5S$. Calculated, %: C 38.09; H 6.39; N 11.10.

D-Xylose 3-sulfanylpropionylhydrazone (Vb). Yield 60%, mp 95–97°C. ^{13}C NMR spectrum (D_2O), δ_C, ppm: **A** (8%): 151.1 (C=N), 169.9 (C=O); **B**, **B′** (30%): 25.4 and 25.8 (CH_2S), 33.4 and 33.7 (**C**H_2CO), 62.8 (C^5), 68.3 (C^1), 69.8 (C^4), 70.3 (C^2), 71.3 (C^3), 172.1 (C=O); α-**C** (10%): 19.1 (CH_2SH), 37.1 (**C**H_2CO), 60.3 (C^5), 68.7 (C^4), 69.3 (C^2), 73.3 (C^3), 86.4 (C^1), 172.5 (C=O); β-**C** (42%): 19.3 (CH_2S), 36.8 (**C**H_2CO), 61.9 (C^5), 68.6 (C^4), 72.2 (C^2), 75.6 (C^3), 89.8 (C^1), 172.6 (C=O). Found, %: C 38.14; H 6.36; N 11.15. $C_8H_{16}N_2O_5S$. Calculated, %: C 38.09; H 6.39; N 11.10.

D-Ribose 3-sulfanylpropionylhydrazone (Vc). Yield 85%, mp 173–176°C. ^{13}C NMR spectrum (D_2O), δ_C, ppm: **A** (10%): 150.6 (C=N), 170.1 (C=O); **B**, **B′** (20%): 26.0 and 26.4 (CH_2S), 33.2 and 33.3 (**C**H_2CO), 63.5 (C^5), 67.2 (C^1), 69.0 (C^4), 70.3 (C^2), 71.6 (C^3), 172.1 (C=O); α-**C** (11%): 18.6 (CH_2SH), 36.8 (**C**H_2CO), 62.4 (C^5), 67.2 (C^4), 68.1 (C^2), 69.5 (C^3), 82.0 (C^1), 172.2 (C=O); β-**C** (59%): 18.8 (CH_2S), 36.5 (**C**H_2CO), 62.5 (C^5), 67.8 (C^4), 70.0 (C^3), 70.2 (C^2), 85.7 (C^1), 172.7 (C=O). Found, %: C 38.06; H 6.42; N 11.17. $C_8H_{16}N_2O_5S$. Calculated, %: C 38.09; H 6.39; N 11.10.

D-Galactose 3-sulfanylpropionylhydrazone (Vd). Yield 65%, mp 174–175°C. ^{13}C NMR spectrum (D_2O), δ_C, ppm: **A** (5%): 152.7 (C=N); 170.2 (C=O); **B**, **B′** (25%): 25.4 and 25.9 (CH_2S), 33.8 and 34.0 (**C**H_2CO), 63.0 (C^6), 66.9 (C^1), 68.7 (C^4), 69.1 (C^5), 69.7 (C^3), 70.6 (C^2), 172.1 (C=O); α-**C** (10%): 19.7 (CH_2SH), 37.2 (**C**H_2CO), 60.8 (C^6), 68.5 (C^3), 70.3 (C^2), 70.5 (C^4), 74.8 (C^5), 87.4 (C^1), 172.4 (C=O); β-**C** (65%): 19.8 (CH_2S), 37.2 (**C**H_2CO), 61.1 (C^6), 69.2 (C^4), 69.8 (C^2), 72.9 (C^3), 75.8 (C^2), 88.9 (C^1), 172.9 (C=O). Found, %: C 38.35; H 6.37; N 9.98. $C_9H_{18}N_2O_6S$. Calculated, %: C 38.29; H 6.43; N 9.92.

D-Glucose 3-sulfanylpropionylhydrazone (Ve). Yield 60%, mp 101–103°C. ^{13}C NMR spectrum (D_2O), δ_C, ppm: **A** (2%): 151.2 (C=N), 170.3 (C=O); **B**, **B′** (10%): 26.2 and 26.6 (CH_2S), 33.9 and 34.0 (**C**H_2CO), 62.5 (C^6), 68.7 (C^1), 70.4 (C^4), 70.8 (C^2), 71.9 (C^3), 73.3 (C^5), 172.1 (C=O); α-**C** (15%): 19.5 (CH_2SH), 37.2 (**C**H_2CO), 60.5 (C^6), 70.0 (C^4), 70.3 (C^5), 70.7 (C^2), 73.8 (C^3), 87.1 (C^1), 172.5 (C=O); β-**C** (73%): 19.7 (CH_2SH), 37.2 (**C**H_2CO), 60.7 (C^6), 69.3 (C^4), 71.2 (C^2), 75.9 (C^5), 76.5 (C^3), 89.3 (C^1), 173.0 (C=O). Found, %: C 38.24; H 6.48; N 9.87. $C_9H_{18}N_2O_6S$. Calculated, %: C 38.29; N 6.43; N 9.92.

D-Mannose 3-sulfanylpropionylhydrazone (Vf). Yield 80%, mp 113–115°C. ^{13}C NMR spectrum (D_2O), δ_C, ppm: **A** (5%): 152.4 (C=N), 170.2 (C=O); **B**, **B′** (20%): 27.0 and 27.2 (CH_2S), 34.2 and 34.3 (**C**H_2CO), 63.1 (C^6), 66.8 (C^1), 68.7 (C^4), 69.1 (C^3), 70.1 (C^2), 70.9 (C^5), 172.1 (C=O); α-**C** (10%): 19.6 (CH_2SH), 37.3 (**C**H_2CO), 60.7 (C^6), 66.5 (C^4), 68.7 (C^3), 72.0 (C^2), 72.5 (C^5), 88.7 (C^1), 172.3 (C=O); β-**C** (65%): 19.8 (CH_2S), 37.3 (**C**H_2CO), 61.2 (C^6), 69.4 (C^4), 70.5 (C^2), 73.2 (C^3), 76.9 (C^5), 87.3 (C^1), 172.5 (C=O). Found, %: C 38.33; H 6.40; N 9.96. $C_9H_{18}N_2O_6S$. Calculated, %: C 38.29; H 6.43; N 9.92.

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