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RING–RING TAUTOMERISM IN THE SERIES OF ISOXAZOLIDINE AND Δ^2-ISOXAZOLINE DERIVATIVES

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We have studied the reaction of 5-hydroxy-3,3,5-trimethylisoxazolidine and 5-hydroxy-3,5-dimethyl-Δ^2-isoxazoline with derivatives of thiosemicarbazide and also thiocarbonohydrazine. Both reactions serve as a method for synthesis of the previously unknown 5-thiosemicarbazido(thiocarbonohydrazino)-isoxazolidines and -Δ^2-isoxazolines. ^{1}H and ^{13}C NMR spectroscopy revealed a tendency of the indicated compounds toward ring–chain and ring–ring tautomeric conversions in solutions involving the 1,2,4-triazolidine, Δ^2-pyrazoline, and 1,2,4,5-tetrazine rings.

Keywords: isoxazolidines, Δ^2-isoxazolines, Δ^2-pyrazolines, tetrahydro-1,2,4,5-tetrazine-2-thiones, 1,2,4-triazolidine-3-thiones, ring–ring tautomerism.

Interest in 5-substituted isoxazolidines and Δ^2-isoxazolines in which the substituent, most often OH or NHR, is directly bonded to the isoxazole ring, is due to the possibility of novel isomeric (tautomeric) conversions, since the presence of the cyclic hemiacetal moiety in such molecules suggests an elevated tendency toward rupture of the $C_{(5)}$–O bond to form linear structures (ring–chain isomerism or tautomerism) [1]. In a number of cases, we may also observe subsequent intramolecular addition of functional nucleophilic moieties at the polar bonds C=O or C=N contained in the resulting linear molecules, and formation of novel cyclic structures (ring–ring isomerism or tautomerism) [2]. This may be illustrated by the examples we observed recently of recyclization of 1-arylpyrimidin-2(1H)-ones to 5-arylureido-Δ^2-isoxazolines when they are treated with hydroxylamine [3] (a typical variant of the ANRORC process [4]), and also ring–ring tautomeric equilibria in solutions of the 5-hydrazino-Δ^2-isoxazoline – 5-hydroxyamino-Δ^2-pyrazoline [5] or isoxazolidine – tetrahydropyrimidine-2(1H)-thione [6] type.

Introduction of a thiosemicarbazide or thiocarbonohydrazine functional group at the 5 position of the isoxazole ring suggests the possibility of complex tautomeric (isomeric) conversions involving additional cyclic forms in addition to isoxazoline (Δ^2-isoxazoline) and linear forms. The tendency toward cyclization of 2(4)-substituted thiosemicarbazones and thiocarbohydrazones to derivatives of 1,2,4-triazolidine-3-thione and tetrahydro-1,2,4,5-tetrazine-2-thione respectively is a general property of compounds in these classes [7-9].

The basic criterion for selecting a specific cyclic structure is the ^{13}C NMR spectroscopy data. Thus for the isoxazolidine (Δ^2-isoxazoline) form, the appearance of a signal from the sp^3-hybridized $C_{(5)}$ atom at 100 ppm (N,C,O-environment) is characteristic [5]. In the five-membered triazolidine ring, the analogous $C_{(5)}$ atom has a chemical shift at 85 ppm (N,C,N-environment) [7], while in the six-membered tetrazine ring, the signal from the

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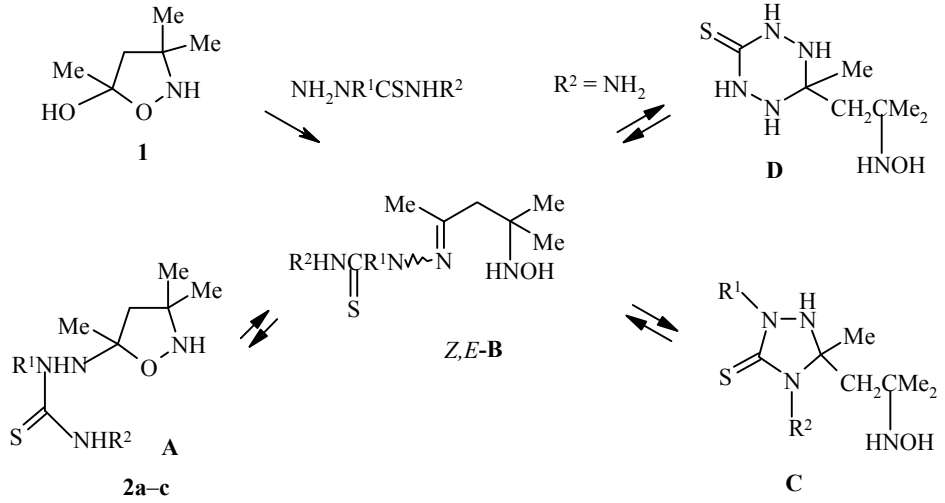
$C_{(6)}$ atom is observed upfield at about 70 ppm [9]. Transition of the cyclic structures to linear structures should lead to disappearance of the "hemiacetal" signals in the 70-100 ppm region and the appearance in the carbon spectra downfield of a signal at 150 ppm (C=N).

Guided by these considerations, we studied the reaction of 5-hydroxy-3,3,5-trimethylisoxazolidine (**1**) with 2(4)-substituted thiosemicarbazides, and also thiocarbonohydrazine.

We found that reaction of the indicated compounds occurs only after prolonged boiling of the reagents in ethanol in the presence of catalytic amounts of acetic acid (see Experimental and Table 1) and leads to formation of the compounds **2a-c** in good yield.



2 a R^1 = H, R^2 = Ph; **b** R^1 = Me, R^2 = Ph; **c** R^1 = H, R^2 = NH_2

Compound **2a** exists in the crystalline state in the cyclic isoxazolidine form **A**, which can be decided from the appearance of a signal from the $C_{(5)}$ atom at 100 ppm in the ^{13}C NMR spectrum taken in the solid phase (Table 2). Dissolution of compound **2a** in DMSO-d_6 leads to the appearance of (in addition to signals from the cyclic form **A**) signals corresponding to the linear form **B**, which is represented by two *Z,E*-configuration isomers. The content of the isoxazolidine form **A** increases as we go from polar solvents to the low-polar $CDCl_3$ (Table 3).

TABLE 1. Physical and Chemical Characteristics of Compounds **2a-c** and **6a-b**

Compound	Form in crystalline state	Empirical formula	Found, % / Calculated, %			mp, °C	Yield, %
			C	H	N		
2a	**A**	$C_{13}H_{20}N_4OS$	55.73 / 55.69	7.22 / 7.19	20.05 / 19.98	134-136	45
2b	**A**	$C_{14}H_{22}N_4OS$	57.16 / 57.11	7.49 / 7.53	19.07 / 19.03	155-157	95
	C	$C_{14}H_{22}N_4OS$	57.07 / 57.11	7.58 / 7.53	18.98 / 19.03	151-153	40
2c	**A**	$C_7H_{17}N_5OS$	38.30 / 38.34	7.76 / 7.81	31.87 / 31.93	142-144	65
6a	**A**	$C_6H_{12}N_4OS$	38.32 / 38.28	6.37 / 6.43	29.80 / 29.76	123-125	50
6b	**A**	$C_6H_{13}N_5OS$	35.41 / 35.45	6.48 / 6.45	34.39 / 34.45	178-180	60

TABLE 2. ^{13}C NMR Spectra of Compounds **2a-c** and **6a,b**

Compound	Solvent	Form	^{13}C NMR spectrum, δ, ppm (25°C, 72 h after dissolution)				
			CH_3	$C_{(3)}$; C–N or C=NOH	$C_{(4)}$ or CH_2	$C_{(5)}$; $C_{(6)}$ or C=N	C=S or $C_{(3)}$
2a	Solid phase	**A**	22.9 ($2CH_3$); 27.1	62.4 ($C_{(3)}$)	52.7 ($C_{(4)}$)	97.2 ($C_{(5)}$)	178.1 (C=S)
	DMSO-d_6	**A**	22.4; 24.9; 27.7	61.4 ($C_{(3)}$)	51.9 ($C_{(4)}$)	99.2 ($C_{(5)}$)	180.7 (C=S)
		***E*-B**	19.2; 23.8; 25.2	57.3 (C–N)	46.3 (CH_2)	154.3 (C=N)	179.5 (C=S)
		***Z*-B**	23.1; 26.4*	56.5 (C–N)	37.6 (CH_2)	154.0 (C=N)	176.8 (C=S)
2b	Solid phase	**A**	23.3 ($2CH_3$); 28.6; 43.9	62.4 ($C_{(3)}$)	53.8 ($C_{(4)}$)	99.7 ($C_{(5)}$)	178.3 (C=S)
	Solid phase	**C**	21.5 ($2CH_3$); 29.4; 38.0	67.3 (C–N)	53.0 (CH_2)	88.4 ($C_{(5)}$)	175.0 ($C_{(3)}$)
	DMSO-d_6*[2]	**A**	21.6; 23.7; 27.0; 42.4	61.0 ($C_{(3)}$)	53.3 ($C_{(4)}$)	100.3 ($C_{(5)}$)	181.6 (C=S)
		C	23.4; 25.7; 27.8; 41.7	66.4 (C–N)	52.6 (CH_2)	87.3 ($C_{(5)}$)	179.3 ($C_{(3)}$)
2c	Solid phase	**A**	23.6 ($2CH_3$); 29.7	61.8 ($C_{(3)}$)	54.1 ($C_{(4)}$)	100.0 ($C_{(5)}$)	181.9 (C=S)
	DMF-d_7	**A**	22.4; 24.1; 27.9	62.0 ($C_{(3)}$)	52.7 ($C_{(4)}$)	100.1 ($C_{(5)}$)	182.9 (C=S)
		***E*-B**	18.4; 25.2*	58.1 (C–N)	46.6 (CH_2)	153.2 (C=N)	178.1 (C=S)
		D	24.7; 26.5*	62.2 (C–N)	40.4 (CH_2)	69.5 ($C_{(6)}$)	172.6 ($C_{(3)}$)
6a	Solid phase	**A**	15.1; 22.1	162.8 ($C_{(3)}$)	46.2 ($C_{(4)}$)	96.4 ($C_{(5)}$)	182.2 (C=S)
	DMF-d_7	**A**	14.6; 21.0	154.5 ($C_{(3)}$)	44.2 ($C_{(4)}$)	97.0 ($C_{(5)}$)	182.8 (C=S)
		E	15.3; 20.5	154.1 ($C_{(3)}$)	46.8 ($C_{(4)}$)	86.7 ($C_{(5)}$)	178.7 (C=S)
6b	Solid phase	**A**	14.5; 24.5	156.4 ($C_{(3)}$)	46.5 ($C_{(4)}$)	97.2 ($C_{(5)}$)	179.9 (C=S)
	DMF-d_7	**A**	13.1; 22.7	156.7 ($C_{(3)}$)	45.7 ($C_{(4)}$)	98.8 ($C_{(5)}$)	180.1 (C=S)
		D	12.0; 21.8	153.6 (C=NOH)	45.3 (CH_2)	75.3 ($C_{(6)}$)	170.6 ($C_{(3)}$)

* Obscured by solvent signals or by signals from the major form.

*[2] Isomer composition of forms A and C.

TABLE 3. Tautomeric Composition and ^{1}H NMR Spectra of Compounds **2a-c** and **6a,b**

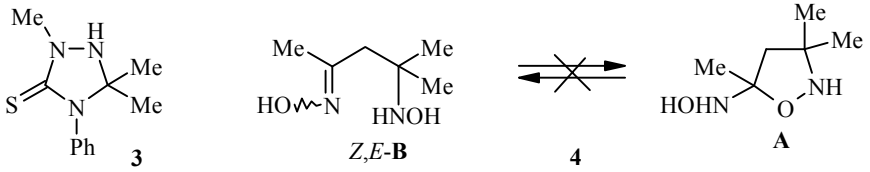
Compound	Solvent	Tautomeric composition, %	^{1}H NMR spectrum, δ, ppm (spin–spin coupling constant, *J*, Hz) (25°C, 72 h after dissolution)		
			CH_3, s	$H_{(4)}$, AB system or CH_2, s	NH or OH, br. s
2a	$CDCl_3$	**A** (90)	1.18 ($2CH_3$); 1.47	1.83; 2.01 (*J* = 13)	5.53; 7.79; 9.01
		E-**B** (10)	1.09 ($2CH_3$); 2.01	2.47 s	4.95; 6.21; 9.14
	DMSO-d_6	**A** (65)	1.11; 1.17;1.38	1.79; 1.94 (*J* = 12)	5.96; 8.75; 9.69
		E-**B** (25)	1.01 ($2CH_3$); 2.03	2.41 s	6.39; 7.01; 9.83
		Z-**B** (10)	1.08 ($2CH_3$); 2.03	2.48 s	7.91;10.25*
2b	$CDCl_3$*[2]	**A** (40)	1.24 ($2CH_3$); 1.46	1.85; 2.12 (*J* = 13)	4.83; 5.51; 9.35
		C (60)	1.59; 1.67; 1.98	2.39; 2.54 (*J* = 13)	4.76; 8.37; 9.22
2c	DMSO-d_6	**A** (70)	1.08; 1.13; 1.31	1.69; 1.87 (*J* = 13)	5.51; 8.66; 9.83
		E-**B** (20)	1.01 ($2CH_3$); 1.93	2.32 s	6.45; 9.02*
		Z-**B** (10)	1.03 ($2CH_3$); 1.93	2.40 s	7.25*
	DMF-d_7	**A** (75)	1.15; 1.22; 1.40	1.81; 2.02 (*J* = 13)	4.38; 5.87; 8.61
		E-**B** (10)	1.10 ($2CH_3$); 2.12	2.47 s	6.78*
		D (15)	1.39; 1.42; 1.47	2.51 s	5.63*
6a	DMF-d_7	**A** (90)	1.52; 1.93	2.82; 2.97 (*J* = 18)	6.11; 7.63; 9.87
		E (10)	1.84; 2.06	3.02 (*J* = 18)*	5.78; 7.86; 8.12
	Pyridine-d_5	**A** (90)	1.63; 1.78	2.81; 3.06 (*J* = 18)	6.34; 9.22; 10.36
		E (10)	1.74; 1.93	2.70; 3.29 (*J* = 19)	5.84; 8.61; 9.71
6b	DMF-d_7	**A** (85)	1.46; 1.87	2.81; 2.93 (*J* = 17)	4.39; 5,91; 8.85
		D (15)	1.38; 1.79	3.27 s	5.94; 9.24
	Pyridine-d_5	**A** (90)	1.57; 1.74	2.74; 3.01 (*J* = 18)	5.25; 7.08; 10.38
		D (10)	1.54; 1.84	3.45 s	5.70; 9.76

* Obscured by solvent signals or signals from the major form.

*[2] Singlet signals from protons of the CH_3N group of forms **A** and **C** are located at 3.68 ppm and 3.73 ppm respectively.

The structure of compound **2b** proved to be interesting: this is the product of condensation of 5-hydroxyisoxazolidine **1** with 2-methyl-4-phenylthiosemicarbazide. Thus during the synthesis, we can isolate a product that has the 1,2,4-triazolidine structure **C** in the crystalline state. This is unambiguously indicated by the presence of a signal at 88.4 ppm ($C_{(5)}$) in the ^{13}C NMR spectrum taken in the solid phase (Table 2). As is completely consistent with the triazolidine structure **C** of compound **2b**, signals are also found in the carbon spectrum for the model 2,5,5-trimethyl-4-phenyl-1,2,4-triazolidine-3-thione (2-methyl-4-phenylthiosemicarbazone of acetone) **3** (see Experimental). The isoxazolidine form **A**, to which correspond two asymmetric $H_{(4)}$ doublets at 1.85-2.12 ppm (AB system) in the NMR spectrum (Table 3 and Fig. 1), accumulates gradually in a solution of compound **2b** in $CDCl_3$. After 14 days, form **A** becomes the only one in solution, and it can be isolated in pure form by removing the solvent. In other words, we observed the recyclization of 1,2,4-triazolidine **C** → isoxazolidine **A** occurring over time.

5-(Thiocarbonohydrazino)isoxazolidine **2c** in a freshly prepared solution in DMSO-d_6, is represented by a tautomeric mixture of cyclic **A** and linear **B** forms, and the latter exists in the form of steric *Z,E*-isomers. The existence in solutions of compounds **2a,c** in the linear form **B**, as generally occurs for most hydrazones tending toward ring–chain tautomeric processes, is explained by stabilization of the hydrazone moiety as a result of *p*,π-conjugation [10]. After 3 days, additional singlet signals from methyl protons at 1.39 ppm, 1.42 ppm, and 1.47 ppm appear in the NMR spectrum of compound **2c** in DMF-d_7, corresponding to one more cyclic form (Table 3) to which, based on comparison of the spectral characteristics with literature analogs [8, 9], should be assigned the tetrahydro-1,2,4,5-tetrazine-3-thione structure **D**. The ^{13}C NMR spectroscopy data (69.5 ppm ($C_{(6)}$) and 172.6 ppm ($C_{(2)}$)) are fully consistent with the proposed cyclic structure of form **D** for compound **2c** (Table 2).



Thus we should note an elevated tendency of compounds **2a-c** toward existence in cyclic forms. In this they are radically different from 4-hydroxyamino-4-methyl-2-pentanone oxime (**4**), their closest structural analog. Compound **4**, as we know [11], in solution completely exists in the linear form **B**, represented by two

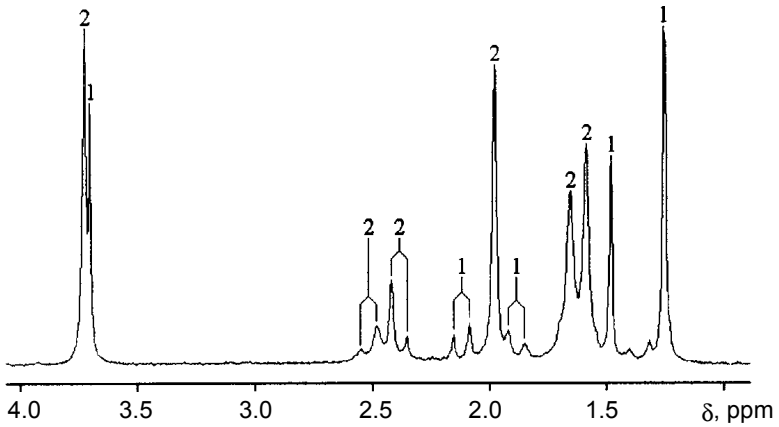


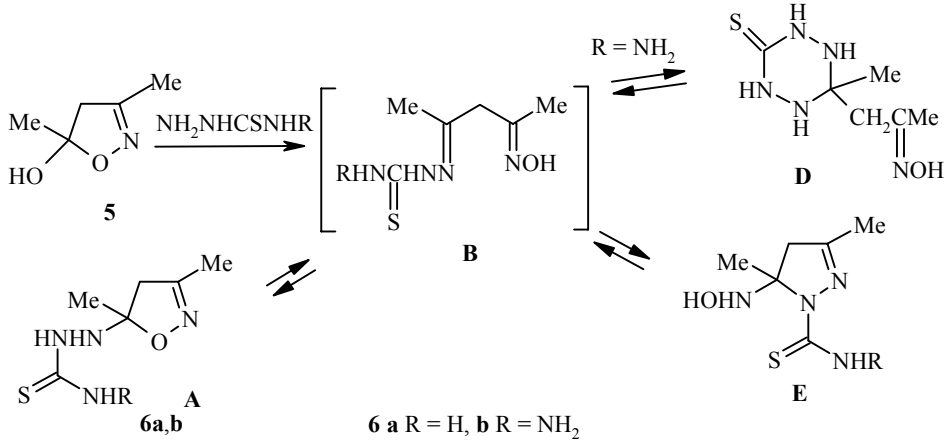
Fig. 1. ^1H NMR spectrum of compound **2b** in $CDCl_3$ (72 h after dissolution):
1) signals from form **A**; 2) signals from form **C**.

configuration isomers, and does not exhibit tendencies toward transition to the alternative 5-hydroxyaminoisoxazolidine form **A**. An analogous tendency toward retention of the oxime moiety has also been noted, by the way, for 1,3-hydroxyamino oximes of other carbonyl compounds [12].

In this regard, 1,3-hydroxyamino oximes are similar to the bisoximes of β-dicarbonyl compounds we studied earlier [13], which can be considered as 5-hydroxyamino-Δ^2-isoxazolines only under special conditions and within very narrow limits. Also note that other 1,3-bifunctional derivatives of β-dicarbonyl compounds (bishydrazones, iminohydrazones (oximes), and hydrazone oximes) exist exclusively in cyclic 5-amino-(hydrazino)-Δ^2-isoxazoline or -Δ^2-pyrazoline forms [3, 5, 14].

Accordingly we should turn our attention to the unanswered question concerning the structure of one more class of bifunctional derivatives of β-dicarbonyl compounds: 1,3-thiosemicarbazone (-thiocarbonohydrazone) oximes. Study of this question is especially important because in this case, it is possible to realize additional tetrahydro-1,2,4,5-tetrazine **D** and Δ^2-pyrazoline **E** forms in addition to the Δ^2-isoxazoline form **A.**

For synthesis of 1,3-thiosemicarbazone(thiocarbonohydrazone) oximes, we carried out the reaction of 3,5-dimethyl-5-hydroxy-Δ^2-isoxazoline **5** with thiosemicarbazide and thiocarbonohydrazide, as a result of which compounds **6a,b** are formed in high yield (see Experimental and Table 1).



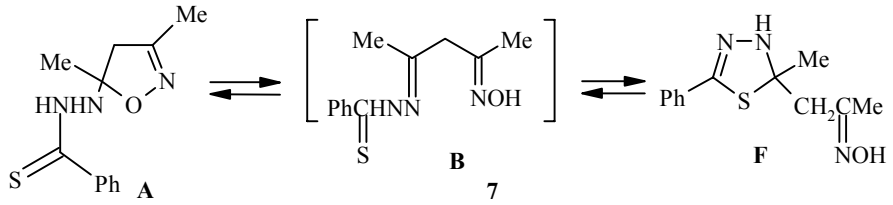
In the crystalline state, compounds **6a,b** exist in isoxazoline form **A**, which is indicated by the signals from the $C_{(5)}$ atoms at 96 ppm and 97 ppm respectively in the ^{13}C NMR spectra taken in the solid phase (Table 2). 3 days after dissolution in pyridine, in the ^1H NMR spectrum of compound **6a**, along with two asymmetric doublets for the $H_{(4)}$ proton forming a typical AB system at 2.80-3.06 ppm, we see an additional AB system indicating the presence of one more cyclic form in solution. The existence of a second cyclic form in solution is also confirmed by the ^{13}C NMR spectrum (Table 2).

The appearance of doubled signals in the ^1H and ^{13}C NMR spectra of compound **6a** can be associated only with a ring–ring tautomeric equilibrium in solution between the isoxazoline form **A** and the pyrazoline form **E**. We already observed recently [5] an analogous variant of tautomeric equilibrium for the example of 1,3-alkanoylhydrazone oximes of acetylacetone, the closest structural analogs of compound **6a**. The spectral characteristics of the previously studied [15] series of 3,5-dimethyl-5-thiosemicarbazido-Δ^2-pyrazolines are also completely consistent with pyrazoline structure **E** of compound **6a**. The tautomeric equilibrium **A** ⇄ **E** is shifted strongly toward the isoxazoline form **A** side, and the percentage of the minor pyrazoline form **E** in all the solvents used (pyridine-d_5, acetone-d_6, DMSO, DMF, acetone, pyridine) does not exceed 5% to 10% (Table 3).

We found that compound **6b** also tends toward ring–ring tautomerism in solution. All the spectral parameters indicate the presence in DMF solution, as in the case of compound **2c**, of the 1,2,4,5-tetrazine-3-thione form **D**. We should note that we did not observe formation in solution of one more possible cyclic form,

the pyrazoline form **E**, which was monitored by taking the ^{1}H NMR spectra of compound **6b** in different solvents with variation of the temperature and time parameters over a broad range.

Thus ring–ring tautomerism is a characteristic feature of 1,3-thiosemicarbazone(thiocarbonohydrazone) oximes of acetylacetone. It is appropriate to recall that the 1,3-thiobenzoylhydrazone oxime of acetylacetone **7** that we studied earlier [16] also is represented in solutions in polar solvents by a tautomeric equilibrium between the isoxazoline form **A** and the 1,3,4-thiadiazoline form **F**. We should note that in all cases, form **A** is predominant in solution. We noted earlier an analogous stability of the isoxazoline ring in an investigation of other functional derivatives of acetylacetone oxime [3, 5, 6].



Thus we have determined synthesis methods for and studied the structure of two new classes of compounds: 5-thiosemicarbazido(thiocarbonohydrazino)isoxazolidines and -Δ^2-isoxazolines. We have noted a tendency of the indicated compounds toward ring–chain and ring–ring tautomeric conversions in solutions involving the 1,2,4-triazolidine, tetrahydro-1,2,4,5-tetrazine, and Δ^2-pyrazoline rings. The data obtained expand our ideas concerning recyclizations (including reversible recyclizations) in the isoxazole derivative series, and supplement our previous research in [3, 5, 6, 17].

EXPERIMENTAL

The ^{1}H NMR spectra were taken on a Bruker AC 200 spectrometer at a frequency of 200 MHz, and the ^{13}C NMR spectra were taken in solutions on a Bruker AM 500 at a frequency of 125 MHz. The high resolution ^{13}C NMR spectra for the crystalline samples were obtained with "magic angle" spinning at a frequency of 3.6 kHz and nuclear cross polarization on a Bruker CXP 100 spectrometer at a frequency of 25 MHz. The number of accumulations of free induction signals was equal to 300. The quantitative composition of the tautomeric forms was determined by integration of the corresponding signals in the ^{1}H NMR spectra. The course of the reactions and the purity of the compounds obtained were monitored by TLC on Silufol UV-254 plates, eluent benzene–acetone, 2:1. The 2-methyl-4-phenylthiosemicarbazide, thiocarbonohydrazine, and also compounds **1** and **5** were obtained by the familiar procedures in [18-21].

5-Thiosemicarbazido(thiocarbonohydrazino)-3,3,5-trimethylisoxazolidines (2a-c) and -3,5-Dimethyl-Δ^2-isoxazolines (6a,b). A mixture of compound **1** (or **5**) (0.02 mol), thiosemicarbazide or thiocarbonohydrazine (0.01 mol), a few drops of acetic acid, and ethanol (50 ml) was boiled for 3 days. After the solvent was removed under reduced pressure, the residue was washed with ether and recrystallized from an ethanol–water mixture, 4:1.

2,5,5-Trimethyl-4-phenyl-1,2,4-triazolidine-3-thione (3). A mixture of acetone (3.0 g, 0.05 mol) and 2-methyl-4-phenylthiosemicarbazide (4.5 g, 0.025 mol) in methanol (50 ml) was boiled for 5 h. After the solvent was removed under vacuum, the residue was recrystallized from benzene. Yield, 4.2 g (75%); mp 132-134°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.37 (6H, s, $2CH_3$); 3.41 (3H, s, CH_3N); 4.45 (1H, br. s, NH); 7.25-7.41 (5H, m, H arom.). ^{13}C NMR spectrum (solid phase), δ, ppm: 21.8 ($2CH_3$); 34.7 (CH_3N); 85.6 ($C_{(5)}$); 127.6-142.5 (C arom.); 180.5 ($C_{(3)}$). Found, %: C 59.73; H 6.78; N 19.04. $C_{11}H_{15}N_3S$. Calculated, %: C 59.69; H 6.83; N 18.99.

REFERENCES

1. R. E. Valters, *Ring–Chain Isomerism in Organic Chemistry* [in Russian] Zinatne, Riga (1978), 238 p.
2. K. N. Zelenin and V. V. Alekseev, *Khim. Geterotsikl. Soedin.*, 825 (1992).
3. A. Yu. Ershov, V. A. Gindin, and A. V. Gribanov, *Zh. Org. Khim.*, **33**, 438 (1997).
4. H. C. van der Plas, *Khim. Geterotsikl. Soedin.*, 1649 (1994).
5. A. Yu. Ershov and A. V. Dobrodumov, *Khim. Geterotsikl. Soedin.*, 852 (2000).
6. A. Yu. Ershov, A. V. Gribanov, V. A. Gindin, and A. I. Kol'tsov, *Zh. Org. Khim.*, **31**, 1054 (1995).
7. O. B. Kuznetsova, V. V. Alekseev, and O. V. Solod, *Modern Methods for Investigation of Organic Compounds* [in Russian], Leningrad State University, Leningrad (1990), p. 32.
8. G. Rajendran and S. R. Jain, *Org. Magn. Resonance*, **22**, 6 (1984).
9. G. Rajendran and S. R. Jain, *Org. Magn. Resonance*, **22**, 550 (1984).
10. Yu. P. Kitaev and B. I. Buzykin, *Hydrazones* [in Russian], Nauka, Moscow (1974), p. 381.
11. A. Belly, F. Petrus, and J. Verducci, *Bull. Soc. Chim. France*, 1395 (1973).
12. A. Ya. Tikhonov and L. B. Volodarskii, *Zh. Org. Khim.*, **9**, 770 (1973).
13. A. Yu. Ershov, S. I. Yakimovich, K. N. Zelenin, and I. V. Zerova, *Zh. Org. Khim.*, **24**, 2287 (1988).
14. A. Yu. Ershov and N. V. Koshmina, *Zh. Org. Khim.*, **34**, 953 (1998).
15. D. H. Hunter, C. McRoberts, and J. J. Vittal, *Can. J. Chem.*, **76**, 522 (1998).
16. K. N. Zelenin, V. V. Alekseev, I. P. Bezhan, A. Yu. Ershov, V. A. Khrustalev, and S. I. Yakimovich, *Khim. Geterotsikl. Soedin.* 1001 (1985).
17. A. Yu. Ershov, *Zh. Org. Khim.*, **31**, 1057 (1995).
18. K. A. Jensen, U. Anthoni, B. Kägi, C. Larsen, and C. T. Pedersen, *Acta Chem. Scand.*, **22**, 1 (1968).
19. L. F. Audrieth, E. S. Scott, and P. S. Kippur, *J. Org. Chem.*, **19**, 733 (1954).
20. C. Harries and L. Jablonsky, *Ber.*, **31**, 1371 (1898).
21. R. Escale, F. Petrus, and J. Verducci, *Bull. Soc. Chim. France*, 725 (1974).