s, 5-H). Mass spectrum, m/z: 167 (M^+, 21), 132 (M^+ –Cl, 100), 105 (46), 79 (11), 69 (14). Compound VIII, PMR spectrum: 4.58 ppm (s, CH_2Cl). Mass spectrum, m/z: 201 (M^+, 20), 170 (15), 166 (M^+ –Cl 100), 105 (50), 79 (16), 69 (14). Compound VI, PMR spectrum: 2.22 ppm (s, CH_3); mass spectrum, m/z: 167 (M^+, 100), 132 (M^+ –Cl, 58), 123 (21), 106 (74), 105 (24), 79 (23), 71 (84), 70 (12), 69 (12). Chlorination of Ie with UV initiation gave similar results.

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MASS SPECTROMETRIC STUDY OF THIOCARBAMOYL-SUBSTITUTED 2-AMINOTHIAZOLES AND 2-IMINOTHIAZOLINES.

1. ALIPHATIC DERIVATIVES

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Electron impact mass spectra of 10 thioureido derivatives of thiazole amino- and iminostructure are studied. The main difference between them appears in the ratio of peak heights of the 2-aminothiazolyl (2-iminothiazoline) and thiazolyl(thiazolinylidene)-2-isothiocyanate ions. A series of decay processes is revealed which occur through rearrangement in the thioureide chain and upon rupture of bonds in the heterocycle.

Fragmentation mass spectrometry in a number of cases represents the most suitable method for identification of isomeric compounds. Among these are 2-amino- and 2-imino- derivatives of benzothiazoles [1, 2], thiazoles [3], and systems analogous to them with other heteroatoms [4]. Knowledge of the similiarities and differences of decay of isomeric compounds is so useful that in some reactions the isomeric composition of the products was impossible to predict earlier [5].

Also, a change in the nature of substituents can complicate interpretation of mass spectra and disrupt the quantitative relation between contributions of characteristic ions. Relative to these questions, we studied mass spectra of a number of thiazole-containing thioureas of general formula

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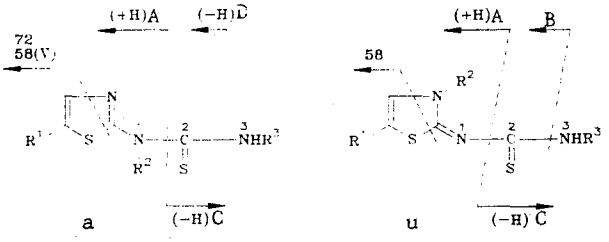


Analogs of these compounds have potent herbicidal, fungicidal, and bactericidal properties [6]. The main purpose of this work was the use of a complex method to reveal the characteristic directions and particular features of decay of this group of compounds, finding a pattern for the distinctive features of isomeric fragmentation (Table 1).

Spectra of all compounds contain molecular ion peaks (M^+). A marked tendency toward increased stability of M^+ (W_M) is observed for the imino derivatives in comparison to the amino derivatives.

As studies of the fragmentation of acylated aminothiazoles [3] and alkoxycarbonyl derivatives of 2-aminobenzothiazole and 2-iminobenzothiazoline [2] showed, the peak intensity ratios of ions which are formed by rupture of the C=S group bonds should be the most characteristic of the isomers studied. Amino derivatives (compounds I, IV-VI, and IX) exhibit the maximal peaks for **A** ions. Rupture of the exocyclic bond $C_{(2)}$–$N_{(3)}$ of these compounds (excluding compound IV) occurs primarily with migration of the $N_{(3)}$ group hydrogen atom onto the neutral fragment with formation of **D** ions (156 and 142*). In the case of the amino derivative IV ($R^3 = CH_3$), migration of the hydrogen atom is less probable, therefore, the analogous 156 ion is not formed. Simple rupture of the $C_{(2)}$–$N_{(3)}$ bond leads to the 171 ion. In the spectra of the imino derivatives II, III, VII, and VIII, peaks of the **B** ions are many times more intense than the **A** ion peaks. Thus, the isomeric forms a and u are readily distinguished mass spectrometrically.

Compounds I-IX are characterized by various susceptibilities toward removal of the sulfur-containing fragments. Thus, molecular ions of amino compounds, except for IV, eliminate a H_2S molecule, and the imino compounds, a HS radical. In the case of IV ($R^2 = CH_3$), a sulfur atom is removed from M^+. The cyclohexyl derivative IX undergoes all three types of decay.

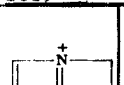
Intense peaks of the rearranged ions $[M - R_3S]^+$ and $[M - R_3SH]^+$ occur in the spectra of the compounds examined (except IV). In the case of I and II, the first of these ions, judging from the defocused metastable spectra (MD), is formed in one step. Consequently, the migration of a methyl radical to the sulfur atom of the thiourea moiety should be important. The ions $[M - CH_3SH]^+$ arise from M^+ as a result of additional migration of a hydrogen atom from the amino group, but a two-step process with elimination of HS and CH_3 ($S + CH_4$) radicals is observed in the MD spectra. Allyl derivatives VI and VII give 139 and 140 ions only as a result of two-step processes. One more transformation is seen in their MD spectra: 179 → 139 (VI) or 180 → 140 (VII). In this case, a C_3H_4 particle is lost. By an analogous mechanism involving elimination of cyclohexene, formation of the 139 and 140 ions from the $[M - H_2S]^+$ and $[M - HS]^+$ ions occurs upon decay of compound IX ($R^3 = c\text{-}C_6H_{11}$).

A relatively intense (~15%) peak for the 125 ion ($C_5H_5N_2S$) is observed in the spectra of imino compounds II and VII. A cyclic structure is probable for this ion (Table 1). It is shifted to m/z 128 in spectra of the CD_3 analogs of III and VIII. The MD spectra of the 125 ions (Fig. 1, a, b) indicates the presence of two alternative two-step decay processes, one of which (stepwise loss of R_3NH and S particles) coincides for both compounds. The second path of formation of the 125 ions occurs as a result of loss of HṠ and CH_2=NH (compound II) fragments or HṠ and CH_2=CH–CH=NH (VII). In both MD spectra, signals indicating synchronous removal of both particles are seen.

Spectra of compounds I-IX contain peaks of ions, the mass numbers of which correspond to R_3NCS^+ ions, however, assignment of these to this type of fragment encounters a number of contradictions. For the imino compounds with $R^3 = CH_3$, besides the component of composition C_2H_3NS, a component of variable contribution, C_3H_5S, occurs. For the imino compound II, this component of 73 ions is insignificant. The main part of them have the composition C_2H_3NS, and would seem to correspond to the **C** ions. However, in the spectrum of the CD_3 an-

*Numbers which characterize ions denote m/z values.

TABLE 1. Mass Spectral Data for Compounds I-IX

Compound	Isomer	m/z (Irel, %)†													
		M^+	W_M	$[M-HS]^+$	$[M-H_2S]^+$	$[M-R_3NCS]^+$ A	$[M-R_3NH]^+$ B	R_3NCS^+ C	$[M-R_3NH_2]^+$ D	$[M-R_3S]^+$	$[M-R_3HS]^+$		72	71	Simple ions‡
I	*a*	187 (50)	10		153 (61)	114 (100)		73 (11)	156 (63)	140 (4)			56	46	99 (C_4H_5NS, 8), 74 (C_2H_4NS, 9), 73 (C_3H_5S, 11), 72 CH_2NCS, 11), 58 (14), 57 (13)
II	*u*	187 (100)	26	154 (45)		114 (12)	157 (59)	73 (16)		140 (5)	139 (2)	125 (15)	10	1	127 ($C_5H_7N_2S$, 9), 99 (C_4H_5NS, 12), 99 ($C_3H_3N_2S$, 4), 74 (C_2H_4NS, 7), 72 (CH_2NCS, 7), 58 (8), 57 (7)
III	*u*	190 (100)	33	157 (40)		117 (4)	160 (50)	73 (3)		143 (3)	142 (2)	128 (12)			130 (7), 102 ($C_4H_2D_3NS$, 7), 102 ($C_3D_3N_2S$, 2), 102 (C_2NS_2, 3), 76 (C_2D_3NS, 4), 76 ($C_3H_2D_3S$, 2), 74 ($C_3H_2D_2S$, 4), 74 (C_2D_2NS, 9), 73 (C_3HD_2S, 3), 72 (C_2H_2NS, 3), 58 (6)
IV	*a*	201 (22)	7			128 (100)	171 (1)	73 (26)	142 (87)				13	10	127 (31), 100 (53), 74 (16), 73 (C_3H_5S, 3), 72 (CH_2NCS, 13), 71 (CHNCS, 5)
V	*a*	199 (73)	10		165 (45)	100 (100)		99 (38)	156 (99)		125 (48)		13	10	85 (C_3H_3NS, 7), 85 (C_2HN_2S, 7), 73 (C_3H_5S, 6), 73 (CH_3NCS, 17), 58 (CNS, 39), 58 (C_2H_2S, 39), 57 (C_2HS, 15), 57 (C_3H_7N, 15)
VI	*a*	213 (76)	11		179 (33)	114 (100)	157 (38)	99 (32)			139 (26)		57	44	198 (47), 73 (17), 72 (C_2H_2NS, 11), 71 (C_2HNS, 6)
VII	*u*	213 (36)	14	180 (5)		114 (7)	157 (100)	99 (18)		140 (9)	139 (4)	125 (13)	9	5	198 (24), 73 (C_3H_5S, 4), 73 (CH_3NCS, 6), 58 (9)
VIII	*u*	216 (35)	13	183 (4)		117 (4)	160 (100)	99 (5)		143 (11)	142 (3)	128 (15)			201 (22), 102 ($C_4H_2D_3NS$, 8), 102 (C_2NS_2, 5), 76 (7), 74 (4), 73 (3), 72 (4), 58 (C_3H_6DN, 1), 58 (C_2H_2S, 6), 57 (C_3H_5DN, 1), 57 (C_2HS, 3)
IX	*a*	255 (59)	20	222 (4)	221 (4)	114 (100)	157 (9)	141 (5)	156 (10)	140 (8)	139 (18)		8	4	179 ($C_{10}H_{13}NS$, 8), 172 ($C_5H_6N_3S_2$, 2), 98 ($C_6H_{12}N$, 13)

*I, IV, VI, IX $R^1 = CH_3$, II, III, V, VII, VIII $R^1 = H$, I, V, VI, IX $R^2 = H$, II, IV, VII $R^2 = CH_3$, III, VIII $R^2 = CD_3$; I-IV $R^3 = CH_3$, V-VIII $R^3 = CH_2{-}CH{=}CH_2$, IX $R^3 = c\text{-}C_6H_{11}$.

†Compound IV $[M - S]^+$ 169 (1); compound IX $[M - S]^+$ 223 (2).

‡Elemental composition is given in parentheses.

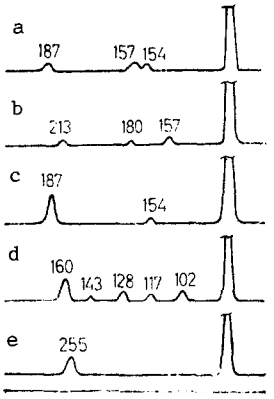
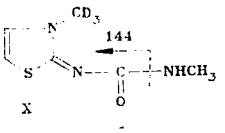


Fig. 1. MD spectra of ions. a) 125 (II); b) 125 (VII); c) 127 (II); d) 76 (III); and e) 179 (IX). The m/z values of the corresponding parent ions are shown over the metastable peaks.

alog of III, the height of the C_2H_3NS ion peak is substantially lower than the height of the C_2D_3NS ion peak (76, Table 1). This implies that for imino compounds of type II and VII the methylisothiocyanate ions can be formed through participation of the methyl substituent on the nitrogen atom of the thiazole ring. In principle, three elemental arrangements are possible for compound III, which consists of the CD_3NCS ion: one due to the NCS group of the thiazole ring and two due to the NCS fragment of the thioureido group upon migration of the CD_3 radical to either of the thioureido nitrogen atoms.

The MD spectrum of the 76 ion (compound III, Fig. 1, d) only emphasizes the multitude of mechanisms of its genesis and does not allow the source of formation of the CD_2NCS fragment to be found since all parent ions revealed can also be parents of the 76 ion of another composition $C_3H_2D_3S$.

The spectrum of a specially synthesized compound, 1-methyl-3-(3-trideuteromethylthiazolinylidene-2)urea (X) was studied for resolution of the question on the production of the CD_3NCS ions and some other fragments.



The property most characteristic of imino derivatives is preserved in this spectrum: the intense peak of the analog **B** ion (144) is much larger than the peak of the analog **A** ion (117, see Experimental). With respect to secondary decay directions, their contributions are substantially redistributed by comparison to the spectrum of compound III. The presence in the spectrum of compound X of CD_2NCS^+ ions and the absence of CD_3NCO^+ ions indicates that the former is formed due to elements of the thiazole ring and the migration process mentioned above of the CD_3 radical to atoms of the thioureido chain is improbable. The height of CH_3NCO^+ and CD_3NCS^+ ion peaks in this spectrum are approximately identical.

Uncertainty exists in evaluation of the contribution of the **C** ions to the total 99 peak height for compounds with $R^3 = CH_2{-}CH{-}CH{=}CH_2$ (VI and VII). In the spectrum of the CD_3 analog of VIII the 99 ion peak height decreases and that of the 102 peak increases. Of the latter, 65% has the composition $C_4H_2D_3NS$. It follows from this that the larger part of the 99 ions which are formed upon decay of imino compound VII have not the R_3NCS^+ structure but the structure of the N-methylazolinium cation-radical with the same elemental composition. Regarding the amino derivative VI, in view of the difficulty of synthesis of the deutero analog, its spectrum was compared with the spectrum of the 5-desmethyl derivative V. The thiazole 85 ion peak was found to have a height 6 times smaller than the 99 ion peak (R_3NCS^+). Consequently, the imine bond should exhibit a larger tendency to rupture than the amine. This is indicated by the presence of yet another type of rearrangement in the starting M^+.

Principal directions in the decay of the heterocycles follow paths leading to formation of 72 ($C_3H_4S^+$) and 71 ($C_3H_3S^+$) ions. Peaks of these ions are more intense for the amino com-

pounds I and VI and the fact that they are shifted to m/z 58 and 57 in the spectrum of V and the spectra of the imino compounds indicates that this group of ions arises upon rupture of the $C_{(2)}$–S and $C_{(4)}$–N bonds of the thiazole ring. In the spectra of the imino compounds, 72 and 71 ion peaks also occur, but their formation is due to a more complex rearrangement process with participation of the methyl radical on the $N_{(3)}$ atom. The presence in the spectrum of the CD_3 analog of III of 76 ($C_3H_2D_3S$), 74 ($C_3H_2D_2S$), and 73 (C_3HD_2S) ions attests to this. Notice that the 73 (C_3H_5S) ion fragments which are analogous in composition to the first of these ions are smaller by comparison with the 72 ion peaks which are recorded in the spectra of amino compounds (I, IV, V, and VII), although for compounds V and VII they can appear due to the allyl group.

The $C_2H_4NS^+$ (74) and $C_2H_2NS^+$ (72) ions are practically everywhere accompanied by methylisothiocyaate 73 ions. Their presence in spectra of the allyl derivatives and the presence of components of the $C_2D_2NS^+$ ion in the spectrum of III indicates that these fragments can also be produced by involvement of the methyl group on ring atom $N_{(3)}$.

We now treat individual features of the spectra of I-IX. In the spectrum of II, the $[M - CH_2NS]^+$ (127) ion peak of composition $C_5H_7N_2S$ occurs. The MD spectrum of this ion (Fig. 1, c) shows that direct elimination of the CH_2NS fragment due to a complex rearrangement is important besides the two-step process of HS and HCN removal. This is analogous to that which determines elimination of CHO_2 upon decay of the methoxycarbonyl derivatives of 2-imino-3-methylbenzothiazoline [2].

Tri-substituted thiourea IV forms the 127 ion by simple rupture of the exocyclic bond $N_{(1)}$–$C_{(2)}$, along with the $[M - R_3NCS]^+$ ion. The former, similarly to the 163 ion in the spectrum of 2-methylaminobenzothiazole [1], decomposes with elimination of an HCN molecule.

All spectra of derivatives with $R^3 = CH_2$–CH=CH_2 contain an intense $[M - CH_3]^+$ fragment peak. The identical form and relative intensity of the metastable peaks corresponding to the $M^+ \rightarrow [M - CH_3]^+$ transition for VI and VII show that the isomerization of M^+ occurs in the allylamine group. The isotopic label is not lost during this process in the spectrum of the deuterium exchange of VI with CD_3OD, consequently, the hydrogen atoms of both amino groups do not migrate to the terminal carbon atom. The reason for formation and stabilization of the $[M - CH_3]^+$ fragments is apparently the isomerization of the allyl group under electron impact, rupture of the CH_3 radical, and closing of the vinyl radical on the sulfur atom of the thiourea.

Spectra of V-IX exhibit peaks for ions of variable intensities R_3NH^+ and $R_3NH_2^+$ at 56 and 57 (V-VIII) and 98 and 99 (IX), respectively. In the spectrum of IX, a fragment peak of composition $C_{10}H_{13}NS$ occurs which is the product of elimination of thiourea from the M^+ with migration of the cyclohexenyl radical to the thiazole ring (Fig. 1, e).

In the spectrum of the urea derivative X, a number of features are observed. The peak of the $[M - NHCH_3]^+$ ion is the base peak. The ions $[M - OH]^+$, $[M - H_2O]^+$, and $[M - CH_3O]^+$ are absent and the $[M - HS]^+$ ion peak has a very low intensity. Consequently, this ion is formed for the thioureido derivatives basically due to the sulfur atom of thiourea. The 100 ion peak of composition C_3H_2NOS in the spectrum of X is more intense than the peak of its analog, the ion of composition $C_3H_2NS_2$ in the spectrum of III. The 126 fragment ($C_4H_2N_2OS^+$) does not have an analog in the spectrum of III and is most probably the thiazolylisocyanate ion. The 86 ion has composition C_2ONS. Since the 102 ion in the case of III and VIII contains a similar C_2NS_2 component, they can be considered to be formed from $-S-\overset{|}{\underset{\cdot}{C}}=NC=O(S)$. Finally, peak of ions, $[M - CD_2NH]^+$ (143) and C_3D_3NS (88), which are formed as a result of unusual rearrangements, are observed in the spectrum of X.

Thus, the ratio of ion peak intensities in the mass spectra of 2-aminothiazole (2-iminothiazoline) and thiazolyl-2-isothiocyanate can be used for discrimination of N-thiazolyl-N'-alkyl(alkenyl)thioureas and their iminoisomers.

EXPERIMENTAL

A MX 1310 mass spectrometer with a SVP5 direct insertion probe was used. The ionization potential was 50 V, collector current was 40 μA, and the temperature of the vaporizer and ionization chamber was 80-90°C. The resolving power was 1200 (low resolution) and 8000 (high resolution, exact mass measurement). Perfluorokerosene was the reference. For defocusing: E, H = const., scanning accelerating potential was 2.0-4.5 kV with a rate of 0.1

TABLE 2. Characteristics of Synthesized Compounds

Compound	Empirical formula	mp, °C	Solvent for recrystallization	Compound	Empirical formula	mp, °C	Solvent for recrystallization
I	$C_6H_9N_3S_2$	146...147	Benzene	IV	$C_7H_{11}N_3S_2$	112...113	Benzene
			Benzene 1:2	VI	$C_8H_{11}N_3S_2$	105...106,5	Alcohol
				VII	$C_8H_{11}N_3S_2$	102...103	Alcohol
II	$C_6H_9N_3S_2$	152...153	Benzene	VIII	$C_8H_8D_3N_3S_2$	102...103	Acetone
III	$C_6H_6D_3N_3S_2$	153...154	Benzene Hexane 1:2	IX	$C_{11}H_{17}N_3S_2$	187...189	Acetone

kV/sec. The frequency of the analyzed compounds was monitored on Silufol UV-254 plates. The elemental analyses for C, H, and N corresponded with those calculated.

General Synthetic Method for Thiazolylthioureas (I, IV-VI, IX). Five mmoles of 2-aminothiazole or its homolog were dissolved in 5-20 ml dry benzene, and 5 mmoles methyl-, allyl-, or cyclohexylisothiocyanate, respectively, were added. The mixture was boiled for 1-8 h and evaporated in air. The dry residue was washed with 5 ml dilute HCl and then water, dried, and recrystallized from the appropriate solvent. Yield 80-96%.

General Synthetic Method for Thiazolinylidenethioureas (II, III, VII, and VIII). Ten mmoles of the iodide hydrate of substituted 2-iminothiazoline were suspended in 10-30 ml dry acetone, an equimolar quantity of triethylamine was added, and 10 mmoles of the corresponding isothiocyanate was added in portions. The mixture was boiled for 1-6 h and the solvent was evaporated in air. The residue was washed with water, dilute HCl, and water again, dried, and recrystallized. Yield 70-98%.

Characteristics of the compounds synthesized are given in Table 2.

1-Methyl-3-(3-trideuteromethylthiazolinylidene-2)urea (X, $C_6H_6D_3N_3OS$. 2-Imino-3-trideuteromethylthiazoline iodide hydrate (0.49 g, 2 mmole) was suspended in 3 ml dry acetone, triethylamine (0.2 g, 2 mmole) and, dropwise with stirring, methylisocyanate (0.12 g, 2.1 mmole) were added. The mixture was boiled for 2 h and evaporated to dryness. The products were separated chromatographically on a silica gel L 100/160 column, eluting with a mixture of acetone–chloroform–benzene, 1:1:1. Yield 0.25 g (72%), mp 94.5-95.5°C. Mass spectrum, m/z (elemental composition; I, %): 174 (M^+, $C_6H_6D_3N_3OS$; 56), 144 ($C_5H_2D_3N_2OS$; 100), 143 ($C_5H_5DN_2OS$; 2) 126 ($C_4H_2N_2OS$; 2), 117 ($C_4H_3D_3N_2S$; 2), 116 ($C_3H_2NS_2$; 1), 116 ($C_4H_2D_3N_2S$; 0.5), 102 ($C_3D_3N_2S$; 0.5), 102 ($C_4H_2D_3NS$; 0.7), 100 (C_3H_2NOS; 5), 88 (C_3D_3NS; 4), 86 (C_2NSO; 2), 76 ($C_3H_2D_3S$; 2), 76 (CD_3NCS; 1), 74 (C_3D_3S; 6), 73 (C_3HD_2S; 2), 72 (C_2DNS; 3), 60 (C_2D_2S; 1), 58 ($C_3H_2D_3N$; 1.5), 58 (C_2H_4NO; 4.5), 58 (C_2H_2S; 3), 57 (C_2H_3NO; 2), 57 ($C_2HD_2N_2$; 2).

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