carbethoxybenzoylchloride [8] for 2 h; the substance precipitated out when the solution was cooled. The yield was 1.1 g (67.3%). A mixed melting sample with substance IV, obtained by the method described above, melted without depression of the melting point. Compounds V and VI were obtained in a similar manner. Substances XVIII, XXI, and XXVI were synthesized by using the ethyl ester of chlorocarbonic acid.

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SYNTHESIS AND RADIOPROTECTIVE ACTIVITY OF S-DERIVATIVES OF MERCAPTOETHYLAMINOMETHYLNAPHTHALENES AND BENZO-2,1,3-THIADIAZOLES

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It is known that substitution of an amino group hydrogen atom in the well-known radioprotective preparation β-mercaptoethylamine or its S-derivatives by bulky substituents results in increased lipoidotropic properties to which the reduced radioprotective dose of the substances obtained is probably related [1].

An analysis of reports in the literature allowed us to conclude that at the present time a tendency is becoming apparent to use compounds belonging to aromatic and heterocyclic systems as substituents, among which substances have been found which are active with regard to radioprotection. For the purpose of seeking out new radioprotectors we synthesized a series of aliphatic sulfoazo-containing compounds (Tables 1 and 2) having benzo-2,1,3-thiazole and naphthalene derivatives as their bulky substituents [2-5].

The requirements for synthesizing 1- and 2-bromomethylnaphthalenes (I and II) and 4- and 5-bromomethylbenzo-2,1,3-thiadiazoles (III and IV) were obtained from reports in the literature [6-8].

When I-IV react with ethyleneimine (see the chart), the corresponding 1- and 2-aziridinylmethylnaphthalenes (V, VI) are formed as well as the 4- and 5-aziridinylmethylbenzo-2,1,3-thiadiazoles (VII, VIII). It should be noted that VII and VIII possess a polymorphism which is expressed in changes in melting point, solubility, and crystalline structure (Fig. 1).

The identity of VII and VIII in their crystalline and amorphous forms is supported spectrophotometrically (Fig. 2) and by chemical methods. The aziridinyls of VII and VIII are broken open by hydrogen bromide with the formation of β-N-(methylbenzo-2,1,3-thiadiazolyl)aminoethylbromides (IX, X). The β-N-(1- and 2-methylnaphthyl)aminoethylbromides (XI, XII) are formed in a similar fashion. Compound IX is isolated as a base, and compounds X-XII in the form of hydrobromides.

When IX reacts with potassium ethylxanthogenate, thiourea, sodium thiosulfate, or ammonium trithiocarbonate, the corresponding xanthogenate (XIII), isothiuronic salt (XIV), Bunte salt (XV), and S,S',N,N',β,β'-bis-(4-methylbenzo-2,1,3-thiadiazolyl)aminoethyltrithio-

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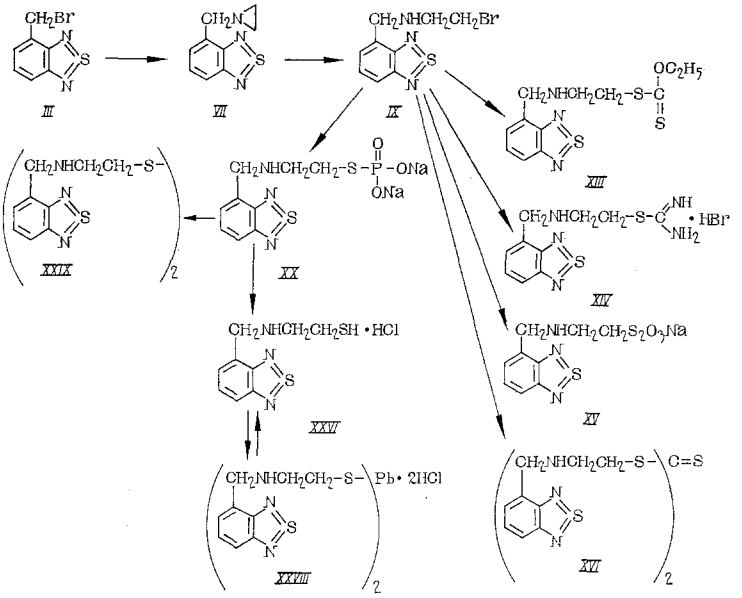


carbonate (XVI) is formed. In a similar manner, when XI is heated in an aqueous solution of sodium thiosulfate, S-β-N(1-methylnaphthyl)aminoethylthiosulfuric acid (XVII) is formed.

When trisodium thiophosphate is reacted with III, IV, IX, X, or XI, the corresponding thiophosphoric acid salts (XVIII-XXII) are formed. The calcium salt of S-β-N-(2-methylnaphthyl)aminoethylthiophosphoric acid (XXIII) was obtained from the bromide of XII.

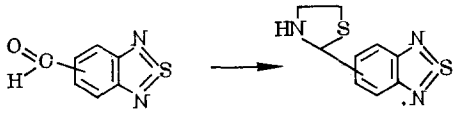
In a hydrochloric acid medium XVIII and XIX are hydrolyzed for 1-2 h to the corresponding 4- and 5-mercaptomethylbenzo-2,1,3-thiadiazoles (XXIV, XXV). Under similar conditions compounds XXII and XXIII barely hydrolyze, and hydrolysis of XX and XXI to β-N-(4- and 5-methylbenzo-2,1,3-thiadiazolyl)aminoethylmercaptans (XXVI, XXVII) takes 48 h.

The mercaptan XXVI forms lead mercaptide (XXVIII) which is converted to XXVI on exposure to hydrogen sulfide. Attempts to obtain XXVI by exposing aziridinyl VII to hydrogen sulfide were not successful. A product of uncertain structure was produced.

The individuality of the thiophosphates XVIII-XXI and mercaptans XXIV-XXVII obtained was established by using thin-layer chromatography.

When the thiophosphates XX-XXIII and mercaptans XXIV and XXV are oxidized by an iodine solution the corresponding disulfides (XXIV-XXXI) are formed.

Subsequent treatment of the 4- and 5-formylbenzo-2,1,3-thiadiazoles (XXX, XXXVI) with ethyleneimine and hydrogen sulfide resulted in the production of the corresponding 2-(4- and 5-benzo-2,1,3-thiadiazolyl)thiazolidines (XXXVII, XXXVIII) which may be regarded as the cyclic analogs of the mercaptans XXVI and XXVII.



Synthesis of the aldehyde XXXVI was accomplished by methods described for XXXV [9] in accordance with the scheme:

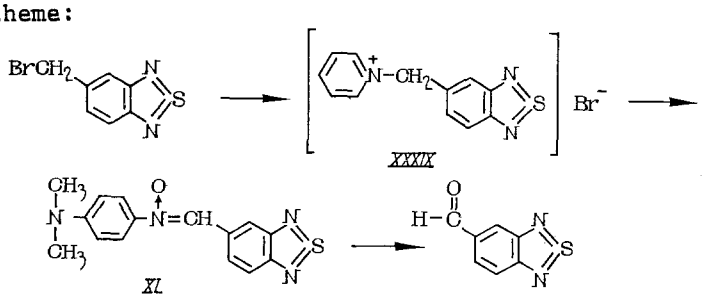
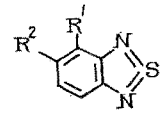
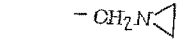
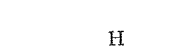


TABLE 1. 4- and 5-Substituted Benzo-2,1,3-Thiadiazoles



Compound	R^1	R^2	Yield, %	Melting point, °C	R_f	Found, %			Empirical formula	Calculated, %		
						N	S	halogen or phosphorus		N	S	halogen or phosphorus
VII		H	79	64—6* 146—1 †	—	22,42	16,75	—	$C_9H_9N_3S$	21,99	16,75	—
VIII	H		90	78—80* 100—8 †	—	21,36	16,27	—	$C_9H_9N_3S$	21,99	16,75	—
IX	$-CH_2NHCH_2CH_2Br$	H	78	262—4	—	15,24	11,12	29,20	$C_9H_{10}BrN_3S$	15,44	11,76	29,41
X	H	$-CH_2NHCH_2CH_2Br \cdot HBr$	83	195—7	—	11,80	8,90	45,44	$C_9H_{10}BrN_3 \cdot HBr$	11,89	9,06	45,32
XVIII	$-CH_2S-\overset{\overset{O}{\|}}{\underset{\underset{ONa}{\|}}{P}}-ONa$	H	97	259 (carbonization temp.)	0,85	6,89	15,37	—	$C_7H_{17}N_2Na_2PO_6S_2$	6,75	15,45	—
XIX	H	$-CH_2S-\overset{\overset{O}{\|}}{\underset{\underset{ONa}{\|}}{P}}-ONa$	90	250 (carbonization temp.)	0,86	6,88	16,18	—	$C_7H_5O_3N_2Na_2S_2P \cdot SH_2O$	7,07	16,21	—
XX	$-CH_2NHCH_2CH_2-S-\overset{\overset{O}{\|}}{\underset{\underset{ONa}{\|}}{P}}-ONa$	H	51	>250	0,60	12,16	18,90	8,85	$C_9H_{10}O_3N_3Na_2 \cdot S_2P_1$	12,03	18,33	8,88
XXI	H	$-CH_2NHCH_2CH_2S-\overset{\overset{O}{\|}}{\underset{\underset{ONa}{\|}}{P}}-OH$	81	>250	0,66	11,09	16,72	8,21	$C_9H_{11}O_3N_3NaPS_2 \cdot 3H_2O$	11,05	16,75	8,15
XXVI	$-CH_2NHCH_2CH_2SH$	H	50	161 (decomp.)	0,58	15,27	23,66	14,31	$C_9H_{11}N_3S_2 \cdot HCl$	16,05	24,48	13,57
XXVII	H	$-CH_2NHCH_2CH_2SH$	62	260—2	0,61	15,80	24,22	13,80	$C_9H_{11}N_3S_2 \cdot HCl$	16,05	24,48	13,57
XXIV	$-CH_2SH$	H	80	40—1	0,67	15,49	34,92	—	$C_7H_6N_2S_2$	15,38	35,16	—
XXV	H	$-CH_2SH$	99	45—6	0,65	15,65	35,46	—	$C_7H_6N_2S_2$	15,38	35,16	—
XXIX	$(-CH_2NHCH_2CH_2-S-)_2$	H	53	194—5		16,21	29,07	—	$C_{18}H_{26}N_6S_4$	18,75	28,52	—
XXX	H	$(-CH_2NHCH_2CH_2-S-)_2$	75	116—7		19,00	29,10	—	$C_{18}H_{20}N_6S_4$	18,75	28,52	—
XXIII	$-CH_2-S-)_2$	H	99	108—10		15,46	34,69	—	$C_{14}H_{10}N_4S_4$	15,49	35,47	
XXXIV	H	$-CH_2-S-]_2$	94	90—2		15,38	34,77	—	$C_{10}H_{10}N_4S_4$	15,49	35,47	

*Crystalline form.
†Amorphous form.

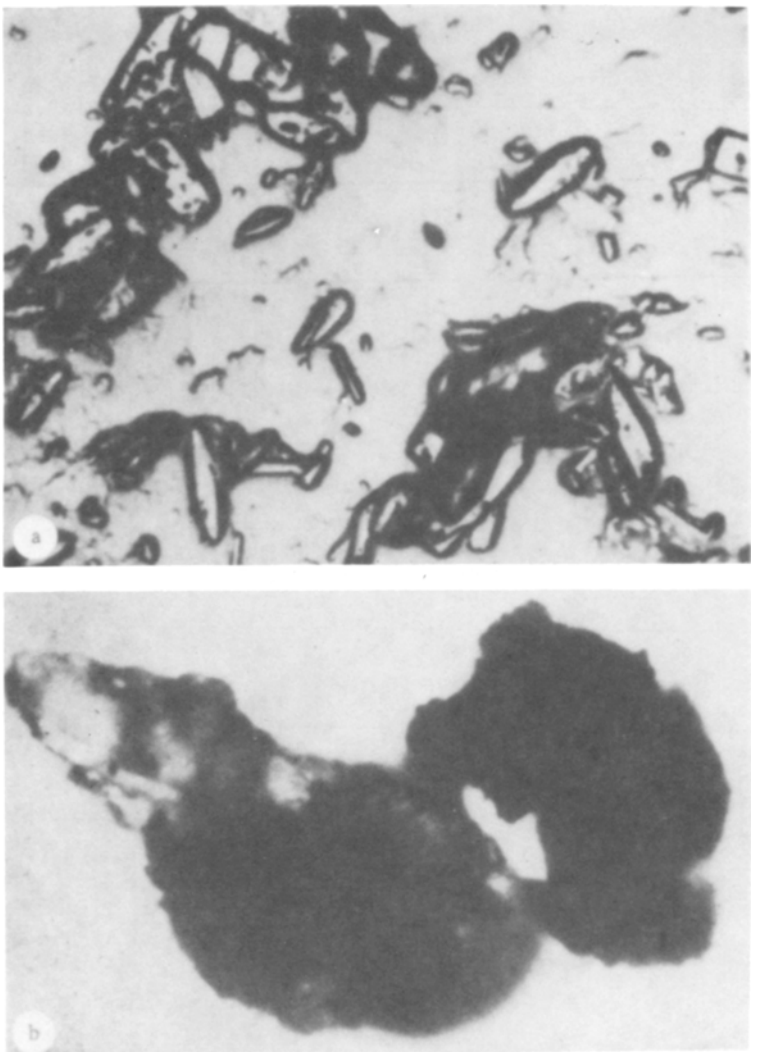


Fig. 1. Structure of VII 5 h after isolation (a) and after 30 days of storage at 20°C (b). Magnification: 113×.

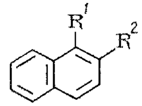
The compounds obtained were checked for toxicity and radioprotective activity in accordance with the methodology described in [2, 3].

It is apparent from Table 3 that 10 of the 15 benzo-2,1,3-thiazole-derived compounds studied have pronounced radioprotective properties. However, substitution of an amino group hydrogen atom in β-mercaptoethylamine and its derivatives by a methylbenzo-2,1,3-thiadiazole radical did not result in a reduction in the radioprotective dose, as was observed, for example, when cyclohexylalkyl derivatives were used [1]. The naphthalene derivatives investigated proved ineffective with regard to radioprotection.

EXPERIMENTAL METHOD

Chromatography of the mercaptans XXIV-XXVII and thiophosphates XVIII-XXI was carried out on Silufol UV-254 sheets. The system concentrated hydrochloric acid—water—methyl alcohol (1:9:10) was used for the mercaptans, and water was used for the thiophosphates. The spots were developed in iodine vapor. The IR-spectra of compounds VII and VIII were taken on spectrophotometer UR-20 in carbon tetrachloride, the UV-spectra were read on apparatus SF-8 in 96% ethanol (approximately 10 M^{-3}). The external appearance of VII and VIII was examined and photographed under microscope MBI-6.

Aziridinyls V-VIII. A mixture of 0.02 moles of I (or II-IV, respectively), 0.03 moles of ethyleneimine, 0.02 moles of triethylamine, and 30 ml of dry benzene were stirred at room temperature for 6-8 h. The precipitate was filtered out, and the filtrate was concentrated in a vacuum. The yield was 75-95% (see Tables 1 and 2).

TABLE 2. 1 and 2-Substituted Naphthalenes 

Yield, %	Melting point, °C	Found, % N	Found, % S	Found, % halogen or phosphorus	Empirical formula	Calculated, % N	Calculated, % S	Calculated, % halogen or phosphorus	Compound	R^1	R^2
98	107—9	7,26	—	—	$C_{13}H_{13}N$	7,65	—	—	V	$-CH_2N\triangleleft$	H
95	90—2	7,80	—	—	$C_{13}H_{13}N$	7,65	—	—	VI	H	$-CH_2N\triangleleft$
73,5	187—9	4,20	—	47,40	$C_{13}H_{14}BrN \cdot HBr$	4,05	—	47,37	XI	$—CH_2NHCH_2CH_2 \cdot Br$	H
66	218—20	3,87	—	45,49	$C_{13}H_{14}BrN \cdot HBr$	4,05	—	46,37	XII	H	$—CH_2NHCH_2CH_2Br$
84	173	5,52	21,94	—	$C_{13}H_{15}NO_3S_2$	4,71	21,54	—	XVII	$—CH_2NHCH_2CH_2S_2O_3H$	H
27	—	3,44	—	—	$C_{13}H_{15}NNaSPO_3 \cdot 2H_2O$	3,94	—	—	XXII*	$—CH_2NHCH_2CH_2—S—P(=P)(—OH)(ONa)$	H
60	—	3,94	10,01	—	$C_{13}H_{14}NSPO_3Ca$	4,18	9,55	—	XXIII †	H	$—CH_2NHCH_2CH_2S—P(=O)(O)_2Ca$
75	176—8	7,02	13,86	—	$C_{26}H_{28}N_2S_2$	6,48	14,81	—	XXXI	$CH_2NHCH_2CH_2—S—)_2$	H
88	205—7	6,03	15,15	—	$C_{26}H_{28}N_2S_2$	6,48	14,81	—	XXXII	H	$CH_2NHCH_2CH_2—S)_2$

*No clearly defined melting point.
†Carbonizes at a temperature above 300°C.

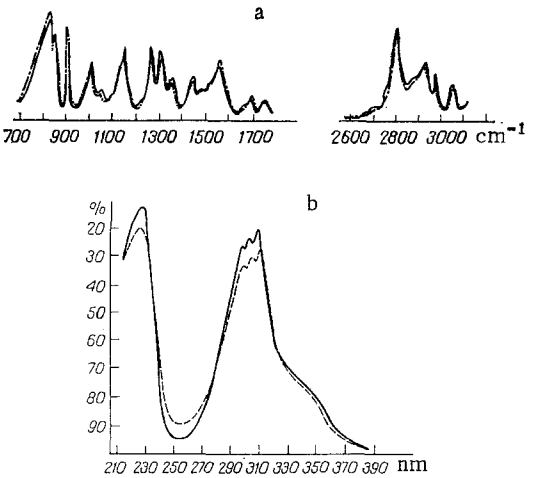


Fig. 2. IR-spectra of a 1% solution of VII in carbon tetrachloride (a) and UV-spectra of a $1 \cdot 10^{-3}$ M solution of VII in ethanol (b). Solid line represents the crystalline form; the broken line represents the amorphous form.

Bromides IX-XII. We dissolved 0.015 mole of the respective aziridinyl V-VIII in 20-75 ml of dry benzene; while cooling on the outside with water, dry hydrogen bromide was bubbled through the solution until it was saturated. The precipitate was filtered out, rinsed with benzene and ether and dried in a vacuum exsiccator. The yield was 65-95%.

S-β-N-(4-methylbenzo-2,1,3-thiadiazolyl)aminoethyl-O-ethyldithiocarbonic Acid (XIII). A mixture of 2.72 g (0.01 moles) of IX, 1.6 g (0.01 moles) of ethylxanthogenate potassium, and 200 ml of isopropyl alcohol was boiled for 5 h, cooled, and the potassium bromide precipitate was filtered out. The product was precipitated out of the filtrate with ether, filtered out, and rinsed with ether. The yield was 0.6 g (20%) XIII, mp: 260°C. Found, %: N 13.01; S 30.65; $C_{12}H_{15}OS_3$. Calculated, %: N 13.42; S 30.67.

S-β-N-(4-methylbenzo-2,1,3-thiadiazolyl)aminoethylisothiuronium Hydrobromide (XIV). A mixture of 2.72 g (0.01 moles) of IX, 0.76 g (0.01 moles) of thiourea, and 200 ml of isopropyl alcohol was boiled for 3 h, cooled, the precipitate was filtered out, washed with isopropyl alcohol and ether, then dried. The yield was 2.8 g (80%) XIV, mp: 224°C (reprecipitation by ether from a methanol solution). Found, %: N 30.01; S 18.70. $C_{10}H_{14}BrN_5S_2$. Calculated: N 30.11; S 18.39.

Sodium Salt of S-β-N-(4-methylbenzo-2,1,3-thiadiazolyl)aminoethylthiosulfuric Acid (XV). A mixture of 2.72 g (0.01 moles) IX, 2.48 g (0.01 moles) of sodium thiosulfate, and 30 ml of water were boiled for 2 h, activated charcoal was added, and it was filtered and cooled. The precipitate was filtered out and dried. The yield was 1.5 g (45%) XV, mp: 187°C (with decomposition, from water). Found, %: N 13.09; S 30.01. $C_9H_{10}O_3NaS_3$. Calculated, %: N 12.84; S 29.35.

S-β-N-(1-methylnaphthyl)aminoethylthiosulfuric Acid (XVII) was obtained from the bromide XI in a manner similar to compound XV. The qualitative test for sodium ion was negative. Yield 84%, mp: 173°C. (with decomposition, from water). Found, %: N 5.52; S 21.94. $C_{13}H_{15}N_3O_2$. Calculated: N 4.71; S 21.54.

S,S',β,β',N,N-(4-methylbenzo-2,1,3-thiadiazolyl)aminoethylthiocarbonate (XVI). To a solution of 5.44 g (0.02 moles) IX in 50 ml of isopropyl alcohol we added a solution of 1.44 g (0.01 moles) of ammonium trithiocarbonate in 5 ml of water and stirred for 5 h; the precipitate was filtered out, washed with alcohol and ether, and dried. The yield was 1.8 g (37%) XVI, mp: 143°C. Found, %: N 17.03; S 32.78. $C_{19}H_{20}N_6S_5$. Calculated, %: N 17.07; S 32.52.

Thiophosphates XVIII-XXVII. We dissolved 0.02 moles of trisodium thiophosphate in a minimum amount of water (20-50 ml) and at 20-40°C added 0.02 moles of the respective bromides (III, IV, IX-XI) dissolved in a minimum amount of isopropyl alcohol, acetone, or water.

TABLE 3. Radioprotective Activity and Toxicity of Several Benzo-2,1,3-Thiadiazole and Naphthalene Derivatives

Compound	Dose, mg/kg	Number of animals	Survival rate, %	P^*	LD_{50}, mg/kg
XIV	40	20	30	>0,05	100
XV	100—150	20	35	>0,05	200
	200	10	60	<0,05	200
XVI	600	10	20	>0,05	900
XVII	100	10	20	>0,05	200
XVIII	300—600	20	40	<0,05	>1000
XIX	600	10	40	<0,05	1200
XX	125	20	40	<0,05	300
	150	10	60	<0,05	300
XXI	150	20	30	>0,05	600
	300	20	85	<0,05	600
XXII	100	10	30	>0,05	300
XXIV	250	30	40	<0,05	800
	500	10	50	<0,05	800
XXV	400	20	35	<0,05	1200
XXVI	100	30	30	<0,05	200
XXVII	100	20	55	<0,05	300
XXIX	100	10	30	>0,05	200
XXX	100	20	45	<0,05	200
XXXI	150	10	20	>0,05	>200
XXXII	150	10	10	>0,05	200
XXXVII	800	10	40	>0,05	>800
XXXVIII	400—800	20	20—30	>0,05	>800

*In relation to an irradiated control.

Stirring followed until the reaction mass was free of SPO_3^{3-} ions (silver nitrate test). The mixture was treated with activated charcoal, filtered, and the filtrate was diluted with 200-300 ml of acetone or isopropyl alcohol. The precipitate which settled out was filtered out, washed with ether or acetone, and dried. Yield 50-90%.

Calcium Salt of S-β-N-(2-methylnaphthyl)aminoethylthiophosphoric Acid (XXIII). To a solution of 3.9 g (0.099 moles) of dodecahydrated trisodium thiophosphate in 20 ml of water we added a solution of 3.45 g (0.01 moles) of XII in 30 ml of water dropwise. It was stirred until the reaction mass was free of SPO_3^{3-} ions (silver nitrate test) and the mixture was decanted into 50 ml of a saturated calcium chloride solution and left in the refrigerator for 24 h. The resultant precipitate was filtered out, washed with water and dried out. The yield was 2 g (60%) XXIII; there was no clearly defined melting point; carbonization occurred at temperatures above 300°C.

Lead Salt of β-N-(4-methylbenzo-2,1,3-thiadiazolyl)aminoethylmercaptan Hydrochloride (XXVIII). To a solution of 0.47 g (0.013 moles) of lead acetate in 30 ml of water at 60°C was added a solution of 0.65 g (0.025 moles) of XXVI dropwise. The resultant precipitate was filtered out, washed with water until free of Pb^{2+} ions, and dried in a vacuum-exsiccator. The yield was 1.3 g (80%) XXVIII; mp: 177°C (in a sealed capillary). Found, %: N 10.92; S 18.20. $C_{18}H_{20}H_6PbS_4 \cdot 2HCl$. Calculated, %: N 11.53; S 17.58.

β-N-(4-methylbenzo-2,1,3-thiadiazolyl)aminoethylmercaptan Hydrochloride (XXVI). A. We dissolved 9 g (0.025 moles) of XX in 100 ml of water, acidified with hydrochloric acid to pH 2.0-3.0 and boiled for 48 h, followed by cooling extraction with chloroform; the solvent was driven off in a vacuum. The yield was 5 g (50%) XXVI which after reprecipitation with ether from methyl alcohol had a mp of 163°C (with decomposition).

B. Hydrogen sulfide was bubbled through a suspension of 2.9 g (0.004 moles) of XXVIII in 200 ml of chloroform at room temperature. The lead sulfide precipitate was filtered out; the filtrate was driven off in a vacuum. The yield was 1.2 g (80%) XXVI; after precipitation from methanol the mp was 163°C (with decomposition) and did not show any depression of the melting point with the substance produced by procedure A.

Mercaptans XXIV, XXV, XXVII. These were obtained in a manner similar to procedure A for XXVI.

Disulfides (XXIX-XXXII). To a solution of 0.01 moles of the respective thiophosphates XX-XXIII in 30 ml of water (or 0.002 moles of mercaptans XXIV or XXV in 20-50 ml of methyl alcohol) a methanol solution of iodine was added dropwise until a nonfading yellow color

appeared. The mixture was diluted with water, the precipitate was filtered out, washed with water and dried, and then purified by reprecipitation from alcoholic aqueous solutions. Yield 53-95%.

2-(4-Benzo-2,1,3-thiadiazolyl)thiazolidine (XXXVII). To a solution of 5 g (0.03 moles) XXXV in 40 ml of methanol at 0°C 1.3 g (0.03 moles) of ethyleneimine was added, after which the reaction mass was saturated with exactly 1 g of hydrogen sulfide by weight. The temperature of the mixture was gradually raised to room temperature, and it was allowed to stand overnight. After driving off the methanol in a vacuum, the residue was dissolved in benzene, and the reaction product precipitated with petroleum ether. The yield was 2 g (16%) XXXVII. Mp: 130°C (with decomposition; sealed capillary). Found, %: N 19.15; S 28.84. $C_9H_9N_3S_2$. Calculated, %: N 18.83; S 28.69.

2-(5-Benzo-2,1,3-thiadiazolyl)thiazolidine (XXXVIII). This is obtained in a manner similar to XXXVII, but using the aldehyde XXXVI. Yield was 45%. Mp: 87°C. Found, %: N 19.02; S 20.10. $C_9H_9N_3S_2$. Calculated, %: N 18.83; S 28.69.

Pyridine Salt of 5-Bromomethyl-2,1,3-thiadiazole (XXXIX). A mixture of 20 g (0.087 moles) of IV and 200 ml of dry pyridine was stirred for 2 h at room temperature. The precipitate was filtered out, washed with alcohol and ether and dried. The yield was 21 g (75%). Mp: 198-199°C. Found, %: N 13.21, 13.39; S 10.45, 10.54; Br 26.51, 26.74. $C_{12}H_{10}H_3SBr$. Calculated, %: N 13.63; S 10.39; Br 25.97.

Nitron (XL). A mixture of 21 g (0.068 moles) of pyridinic salt, 8 ml of piperidine, 10.2 g (0.068 moles) of p-nitrosodimethylaniline, and 300 ml of methanol were kept at room temperature for 96 h; the precipitate was filtered out, carefully washed with ethanol and ether and dried. The yield was 17 g (83%). Mp: 185°C (with decomposition). Found, %: N 18.23, 17.93; S 9.80, 9.85. $C_{15}H_{14}N_4OS$. Calculated, %: N 18.12; S 10.73.

5-Formylbenzo-2,1,3-thiadiazole (XXXVI). A mixture of 21 g (0.07 moles) of XL, 500 ml of 15% hydrochloric acid, and 300 ml of chloroform was vigorously stirred for 30 min at room temperature. The chloroform layer was separated and evaporated till dry in a vacuum. Yield: 8.2 g (88%). Mp: 94°C (reprecipitation with water from a methanol solution). Found, %: N 17.15, 16.97; S 19.01, 18.87. Calculation, %: N 17.07; S 19.51.

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