SYNTHESIS OF 2,4-DISUBSTITUTED THIAZOLES

M. Lozinskii, S. Kukota, P. Pel'kis, and T. Kutrya

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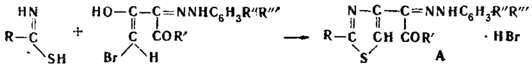
By the reaction of thiourea, thioacetamide, and thiobenzamide with arylhydrazones of γ-bromoacetic ester [1], with boiling for several hours in absolute ethanol, we have obtained previously unreported thiazolium salts (table)



When the salts IV and VI were recrystallized from aqueous ethanol, the free bases were obtained. The reaction of IV with hydrazine hydrate in absolute ethanol gave the hydrazide VIII, which by condensation with phenyl isothiocyanate, was converted into the corresponding phenyl thiosemicarbazide **IX**.

The structure A was confirmed by the IR absorption spectra. The IR spectra have the characteristic frequencies for C=O (1700–1675 cm^{-1}) and C=N (1620–1580 cm^{-1}) and bands of a disubstituted thiazole ring (1340–1300, 1080–1040, 970–930, 880–700 cm^{-1}) [2, 3].

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Institute of Organic Chemistry AS UkrSSR, Kiev

2,4-Disubstituted Thiazole Derivatives*

Compound	R**	R″	Mp, °C***	Empirical formula	Found, %				Calculated, %				Yield, %
					C	H	N	S	C	H	N	S	
I	NH_2	o-CH_3O	197—199	$C_{14}H_{16}N_4O_3S \cdot HBr$	42.37	4.16	—	7.80	41.90	4.27	—	7.99	97
II	NH_2	p-CH_3O	168—169	$C_{14}H_{16}N_4O_3S \cdot HBr$	42.23	4.24	14.17	7.56	41.90	4.27	13.96	7.99	69
III	NH_2	o-C_2H_5O	190—190.5	$C_{17}H_{22}N_4O_4S \cdot HBr$	43.88	5.04	—	6.60	44.44	5.15	—	6.98	56
IV	CH_3	p-CH_3O	72—73	$C_{15}H_{17}N_3O_3S$	—	—	13.02	9.61	—	—	13.15	10.03	74
V	CH_3	p-CH_3	138—143 (decomp.)	$C_{15}H_{17}N_3O_2S \cdot HBr$	—	—	10.47	8.20	—	—	10.93	8.34	64
VI	C_6H_5	p-CH_3O	105	$C_{20}H_{19}N_3O_3S$	—	—	11.13	8.23	—	—	11.01	8.40	53
VII	C_6H_5NH	H	190—192	$C_{19}H_{18}N_4O_2S \cdot HBr$	—	—	12.56	7.15	—	—	12.52	7.16	69
VIII	CH_3	p-CH_3O	158—160	$C_{13}H_{15}N_5O_2S$	—	—	22.81	10.35	—	—	22.93	10.50	100
IX	CH_3	p-CH_3O	180 (decomp.)	$C_{20}H_{20}N_6O_2S_2$	—	—	18.95	14.42	—	—	19.07	14.55	70

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*I, II, IV–IX R‴ = H; III R‴ = 5-OC_2H_5.

**I–VII R′ = OC_2H_5; VIII R′ = $NHNH_2$; IX R′ = $NHNHCSNHC_6H_5$.

***I–III, V, and VII were recrystallized from absolute ethanol, VIII from a mixture of ethanol and dioxane (1:2), and IX from a mixture of ethanol and acetone (1:1).

REACTION OF BENZIMIDAZOLE AND 2-METHYLBENZIMIDAZOLE WITH 1-NITROPROP-1-ENE

V. P. Dukhovskoi, V. D. Tyurin, and G. A. Shvekhgeimer

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1-Nitroalkyl derivatives of benzimidazole have not been described in the literature. We have obtained the previously unknown 1-(β-nitroisopropyl)benzimidazole (**Ia**) and 1-(β-nitroisopropyl)-2-methylbenzimidazole (**Ib**) by the addition of 1-nitroprop-1-ene to benzimidazole and 2-methylbenzimidazole, respectively. By the action of zinc and hydrochloric acid, **Ia** was reduced to 1-(β-aminoisopropyl)benzimidazoles (**II**).