UDC 547.743.1

SYNTHESIS OF PYRROLIDINES, PYRROLINES, AND PYRROLES

XV. Synthesis of Some Sulfamide Derivatives Based on α-(2-Chloroethyl)-β-chlorocrotonaldehyde*

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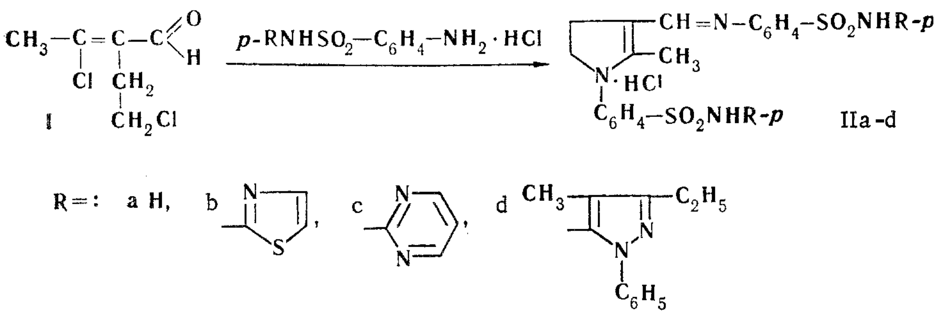
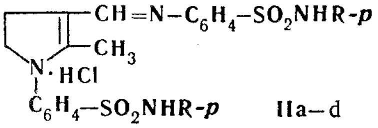
As compounds of potential physiological activity, new sulfamide derivatives, Schiff's bases derived from N-arylpyrrolines, are obtained by reacting sulfanilamide, 2-(p-aminobenzenesulfamide) thiazole, 2-(p-aminobenzenesulfamido)-pyrimidine, and 1-phenyl-3-ethyl-4-methyl-5-(p-aminobenzenesulfamido) pyrazole with α-(2-chloroethyl)-β-chlorocrotonaldehyde.

Continuing research on α-(2-chloroethyl)-β-chlorocrotonaldehyde (I) [2], it has now been reacted with concentrated solutions of sulfanilamide, 2-(p-aminobenzenesulfamido) thiazole, 2-(p-aminobenzenesulfamido) pyrimidine, and 1-phenyl-3-ethyl-4-methyl-5-(p-aminobenzenesulfamido) pyrazole in hydrochloric acid, to obtain new sulfamide preparations which are Schiff's bases derived from N-arylpyrrolines (IIa-d)



The compounds prepared are at present being tested for pharmacological activity.

Compounds Having the General Formula



Compound No.	mp, ° C (decomp)	Formula	Found, %			Calculated, %			Yield, %
			C	H	N	C	H	N	
II a	254	$C_{18}H_{20}N_4O_4S_2 \cdot HCl$	47.09 47.09	4.70 4.58	12.12 12.01	47.32	4.63	12.26	Quantitative
II b	226—227	$C_{24}H_{22}N_6O_4S_4 \cdot HCl$	46.16 45.96	3.70 3.84	13.40 13.28	46.25	3.72	13.49	Quantitative
II c	244	$C_{26}H_{24}N_8O_4S_2 \cdot HCl$	51.06 50.74	4.04 4.29	18.34 18.56	50.93	4.11	18.28	97
IId	216—217	$C_{42}H_{44}N_8O_4S_2 \cdot HCl$	61.13 60.88	5.86 5.51	13.45 13.17	61.09	5.50	13.58	70

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* For Part XIV see [1].

Experimental

α-(2-Chloroethyl)-β-chlorocrotonaldehyde (I) was added (molar ratio of I : sulfamide = 1 : 2.1) dropwise, with shaking, to a concentrated solution of sulfanilamide, 2-(p-aminobenzenesulfamido)-thiazole, 2-(p-aminobenzenesulfamido) pyrimidine, or 1-phenyl-3-ethyl-4-methyl-5-(p-aminobenzenesulfamido) pyrazole in HCl (1:1). After all of I had been added, shaking was continued for 30–40 min longer, and the reaction mixture kept for 1–1.5 days at room temperature. A yellow precipitate formed. Compound IId separated as a crystalline precipitate when the reaction products were diluted with water after reaction was complete. The precipitate was filtered off, washed with ether, and recrystallized from aqueous ethanol containing a few drops of HCl. For analysis the substance was vacuum-dried over P_2O_5.

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RESEARCHES ON 2, 1, 3-THIA- AND SELENADIAZOLE

XXXIX. Polymorphism of 4-Hydroxybenzo-2,1,3-thiadiazole and its Methyl Derivatives*

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4-Hydroxy-, 4-hydroxy-5-methyl-, 4-hydroxy-7-methylbenzo-2,1,3-thiadiazoles are polymorphous.

4-Hydroxybenzo-2,1,3-thiadiazole (I), 4-hydroxy-5-methyl- and 4-hydroxy-7-methylbenzo-2, 1, 3-thiadiazoles (II and III) melt at 114–115°, 110–112°, 100–102° C, respectively, after recrystallization from water [2-4], but after recrystallization from petrol ether [5] they melt at 128–129°, 124–125°, and 119–120° C [5]. In this connection we recrystallized these phenols repeatedly from petrol ether after recrystallizing them from water, and their melting points rose as expected [5]. On the other hand, the compounds with melting points 128–129°, 124–125°, 119–120° C (ex petrol ether), after repeated crystallization from water melted at 114–115°, 110–112°, 100–102° C, respectively.

IR spectra,** measured in vaseline, with a UR-10 instrument and elementary analyses showed the identities of the compounds, consequently it can be concluded that the phenols in question are polymorphous.

It is of interest that 5-hydroxybenzo-2,1,3-thiadiazole (mp 156–157° C) and 5-hydroxy-4-methylbenzo-2,1,3-thiadiazole (mp 121–123° C) are not altered in melting point when recrystallized from water or benzene ether.

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*For Part XXXVIII see [1].

**Spectra determined by V. S. Korobkov.