SYNTHESIS OF SOME 5-PYRIMIDINESULFONAMIDES

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From uracil- and 4-methyluracil-5-sulfonyl chlorides, nine previously unknown 5-pyrimidinesulfonamides have been synthesized.

The potentially biologically active 5-pyrimidinesulfonamides (I–X) were obtained by the reaction of uracil- and 4-methyluracil-5-sulfonyl chlorides [1–3] with various amino compounds. The latter were bis-(β-chloroethyl)amine, δ-diethylamino-α-methylbutylamine, and dimethyl-1,1-biguanide in the form of the hydrochlorides, and dicyandiamide, semicarbazide, and thiosemicarbazide. The reactions were carried out in anhydrous solvents in the presence of an equimolecular amount of triethylamine.

The melting points, yields, and analyses of the substances obtained are given in the table.

EXPERIMENTAL

The appropriate amine and 2–3 drops of triethylamine were dissolved in an anhydrous solvent, then the sulfonyl chloride and triethylamine were added alternately, the medium being kept weakly alkaline. The solvent was evaporated in vacuum and the residue was treated with dry solvents until a crystalline mass was obtained, which was dried in a vacuum desiccator. In those cases where a precipitate of triethylamine hydrochloride formed, it was filtered off before the distillation of the solvent. Compounds I and VI were obtained in dioxane, II and VII in dichloroethane, and III–V and VIII–X in dimethylformamide. In the synthesis of compounds II, VI, VII, and X the starting materials were charged at 10–15° C and left at 20° C for 40–42 hr; in the synthesis of II–VII, the reaction took place practically instantaneously. In the synthesis of I, III, IV, V, VIII, and IX after the reactants had been mixed at 20° C, the mixture was kept at 50–60° C for 4–5 hr.

REFERENCES

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3. I. Kh. Fel'dman and D. I. Biskupskaya, ZhOKh, collection: Synthesis of Natural Compounds and their Analogs and Fragments [in Russian], p. 266, 1965.

4. German Federal Republic patent no. 1 023 757, 1958; C., **129**, 7895, 1958.

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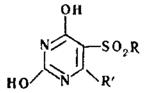
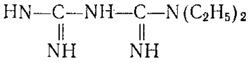
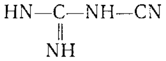
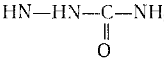
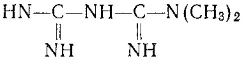
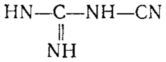
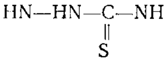


Compound	R	R′	Mp, (decomp.), °C[a]	Empirical formula	Found, % N	Found, % S	Found, % Cl	Calculated, % N	Calculated, % S	Calculated, % Cl	Yield, %
I	$N(CH_2CH_2Cl)_2$	H	275.5—276	$C_8H_{11}Cl_2N_3O_4S$	13.30	10.31	22.65	13.29	10.12	22.47	70
II	$HNCH(CH_3)(CH_2)_3N(C_2H_5)_2$	H	156[b]	$C_{13}H_{24}N_4O_4S \cdot HCl$	15.01	8.57	9.44	15.19	8.68	9.63	100
III	 $HN{-}\underset{\underset{NH}{\Vert}}{C}{-}NH{-}\underset{\underset{NH}{\Vert}}{C}{-}N(C_2H_5)_2$	H	251—252	$C_8H_{13}N_7O_4S \cdot HCl$	29.18	9.56	10.76	28.86	9.42	10.45	81
IV	 $HN{-}\underset{\underset{NH}{\Vert}}{C}{-}NH{-}CN$	H	above 300	$C_6H_6N_6O_4S$	32.68	12.16	—[c]	32.55	12.40	—[c]	75
V	 $HN{-}HN{-}\underset{\underset{O}{\Vert}}{C}{-}NH_2$	H	220—221	$C_5H_7N_5O_5S \cdot H_2O$	26.70	11.82	—	26.20	11.99	—	90
VI	$N(CH_2CH_2Cl)_2$	CH_3	255—255.5[d]	$C_9H_{13}Cl_2N_3O_4S$	12.69	9.67	21.61	12.72	9.70	21.51	56
VII	$HNCH(CH_3)(CH_2)_3N(C_2H_5)_2$	CH_3	269—270[b]	$C_{14}H_{26}N_4O_4S \cdot HCl$	14.47	8.02	9.12	14.63	8.36	9.28	100
VIII	 $HN{-}\underset{\underset{NH}{\Vert}}{C}{-}NH{-}\underset{\underset{NH}{\Vert}}{C}{-}N(CH_3)_2$	CH_3	194—195	$C_9H_{15}N_7O_4S \cdot HCl$	28.13	8.88	10.51	27.72	9.05	10.04	71
IX	 $HN{-}\underset{\underset{NH}{\Vert}}{C}{-}NH{-}CN$	CH_3	above 300	$C_7H_8N_6O_4S$	30.48	11.48	—	30.88	11.76	—	70
X	 $HN{-}HN{-}\underset{\underset{S}{\Vert}}{C}{-}NH_2$	CH_3	203—204	$C_6H_9N_5O_4S \cdot H_2O$	23.50	21.17	—	23.56	21.54	—	92

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[a] I and VI from absolute ethanol; III, IV, and VIII precipitated from absolute methanol with ether; II, V, and VII precipitated from water with ethanol; IX from 70% ethanol; X from water. [b] Very hygroscopic. [c] Found, %: C 27.92; H 2.21. Calculated, %: C 27.86; H 2.32. [d] 263–264 [1].