CARBON SUBOXIDE AND SOME OF ITS REACTIONS

XXVIII. Reaction Of Carbon Suboxide With 2-Amino-1,3,4-thiadiazoles

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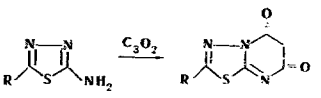
The reaction of carbon suboxide with a number of 2-amino-1,3,4-thiadiazoles has given the corresponding pyrimido[1,2-b]-1,3,4-thiadiazoles

In order to broaden the possibility of the synthesis of various condensed pyrimidoheterocyclic systems from carbon suboxide, we have studied the reaction of the latter with 2-amino-1,3,4-thiadiazole and a number of its derivatives.

It has been established that this reaction takes place with closure of the pyrimidine ring to form, respectively, pyrimido[1,2-b]thiadiazole-1,3-dione and its 6-alkyl or 6-aryl derivatives (table).



N, N'-Malonylbis-2-aminothiadiazoles, which might also be expected by analogy with previous work [1–4] were not found in the reaction products.

The structure of the reaction products was confirmed by their IR spectra, in which there are two bands in the region of carbonyl absorption, which shows the presence of β-dicarbonyl groupings, and, in addition, there is no absorption in the 3450–3200 cm^{-1} region characteristic for an NH group.

EXPERIMENTAL

Gaseous carbon suboxide was obtained by the method described previously [5] and was passed into the reaction mixture directly from the pyrolysis furnace.

Pyrimido[1,2-b]-1,3,4-thiadiazole-1,3-diones (table). At 55–60° C, a 1.5-fold excess of carbon suboxide was passed through a solution of 1.0 g of 2-amino-1,3,4-thiadiazole or one of its 5-derivatives in 60 ml of acetone. As the C_3O_2 was passed in, a crystalline precipitate deposited, and, after standing overnight, was filtered off and washed with ether. It was recrystallized from ethanol. The products were sparingly soluble in water and in organic solvents but solvent in dilute alkalies.

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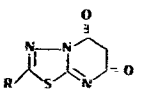
Pyrimido [1,2-b]-1,3,4-thiadiazole-1,3-diones



R	Mp, ° C (decomp)	Empirical formula	Found, %		Calculated, %		Yield, %
			N	S	N	S	
H	Decomp. above 200	$C_5H_3N_3O_2S$	24.71 24.76	19.05 19.00	24.85	18.93	93
CH_3	249—250	$C_6H_5N_3O_2S$	22.70 22.75	17.44 17.69	22.95	17.43	92
C_2H_5	219—221	$C_7H_7N_3O_2S$	21.58 21.34	15.95	21.32	16.24	87
C_6H_5	269—270	$C_{11}H_7N_3O_2S$	16.92 16.99	13.53 13.27	17.14	13.06	93
p-BrC_6H_4	270—271	$C_{11}H_6N_3O_2SBr$	13.20 13.14	9.99 9.72	12.96	9.87	96
p-$MeOC_6H_4$	251—253	$C_{12}H_9N_3O_3S$	15.30 15.48	11.30 11.71	15.27	11.63	94
o-$O_2NC_6H_4$	237—239	$C_{11}H_6N_4O_4S$	19.22 19.12	10.93 10.86	19.31	11.03	92
$C_6H_5CH=CH$	268—269	$C_{13}H_9N_3O_2S$	15.64 15.69	11.91 10.93	15.50	11.80	90