3-Chlorosulfonyl-6-methyl-2 H-1,3,5-oxadiazine-2,4(3 H)-dione

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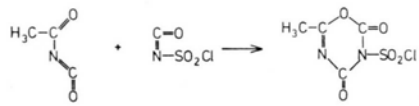
Reaction of acetylisocyanate with chlorosulfonylisocyanate leads to 3-chlorosulfonyl-6-methyl-2 H-1,3,5-oxadiazine-2,4(3 H)-dione.

3-Chlorosulfonyl-6-methyl-2 H-1,3,5-oxadiazine-2,4(3 H)-dione has been obtained from the reaction of acetyl-isocyanate with chlorosulfonylisocyanate at 70 °C for half an hour. The results of NMR and mass spectra of the compound have been reported.



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Ulrich described the reaction of acetylthiocyanate with methylisocyanate[1]. Analogous to the preparation of 2,5-dimethyl-$\Delta^{2,3}$-oxadiazine-4,6-dione[2] and 2-methyl-5-phenyl-$\Delta^{2,3}$-oxadiazine-4,6-dione[3], 3-chloro-sulfonyl-6-methyl-2 H-1, 3,5-oxadiazine-2,4-(3 H)-dione has been prepared by reacting acetylisocyanate and chlorosulfonylisocyanate. The reaction was carried out at 70 °C and after thirty minutes yielded a pale yellow crystalline compound (decomp. at 85 °C). The NMR spectrum of **1** was recorded in dimethylsulfoxide showed a signal at $\tau = 7.7$ ppm for methyl protons. The IR spectrum in KBr showed the usual absorptions of CO$^-$, SO$^-$, C–O–C and S–Cl$^-$ groups at 1720 cm^{-1}, 1160 cm^{-1}, 1050 cm^{-1} and 540 cm^{-1} respectively. The mass spectrum gave intense peak at *m/e* 99 and 127 representing $ClSO_2$ and $C_4H_3N_2O_3$ respectively.

Experimental

1. Preparation of acetylisocyanate[4]

2. Preparation of $C_4H_3ClN_2O_5S$ (**1**): 1.7 g (0.02 mole) of acetylisocyanate and 2.8 g (0.02 mole) chlorosulfonylisocyanate were heated without solvent at 70 °C for half an hour. 3-Chlorosulfonyl-6-methyl-2 H-1,3,5-oxadiazine-2,4-(3 H)-dione crystallizes on cooling (yield 1.75 g, 40%). The substance was dried by washing with sodium dried ether.

Found
C 22.03 H 1.47 Cl 15.48 N 12.25 S 14.31,

Calcd
C 21.24 H 1.32 Cl 15.48 N 12.38 S 14.15.

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[1] H. ULRICH, "Cycloaddition Reactions of Heterocumulenes," Table 12, Academic Press, New York 1967; see also J. GOERDELER and H. SCHENK, Chem. Ber. **98**, 2954 and 3831 [1965].
[2] N. SINGH and P. LATSCHA, Z. Naturforsch. **25b**, 1180 [1970].
[3] N. SINGH and P. LATSCHA, Curr. Sci. **47**, 176 [1972].
[4] O. C. BILLETER, Berl. Ber. **36**, 3213 [1903].