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Interesting Errors in Sulfur Chemistry, 4

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INTERESTING ERRORS IN SULFUR CHEMISTRY, 4

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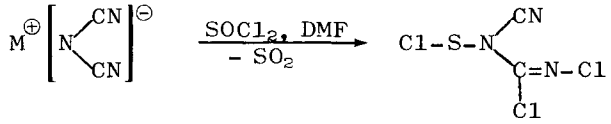
CHLOROTHIO(DICHLOROFORMAMIDE)CYANAMIDE

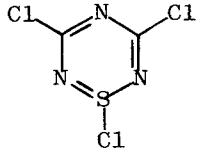
In 1970 Geevers *et al.*[1] described the reaction of the sodium or tetramethylammonium salt of dicyanoamide with thionyl chloride in the presence of dimethylformamide to chlorothio(dichloroformamide)cyanamide **1** in yields of up to 50–65%.



$M^{\oplus}$ = Na; $(CH_3)_4N$ **1**

Evidence for the structure of **1** was presented and the unexpected course of the reaction discussed. The CA name of **1** is N-chloro-N-chlorothio-N-cyano-carbamimidic chloride, CAS registry No. [25816-28-83].

Later in 1974 by Schramm *et al.*[2] the compound originally regarded as **1** was shown by ^{13}C NMR, ESCA, IR, and crystal structure determination[3] (compound **3**) to possess the structure of 1,3,5-trichloro-1λ⁴,2,4,6-thiatriazine **2.**



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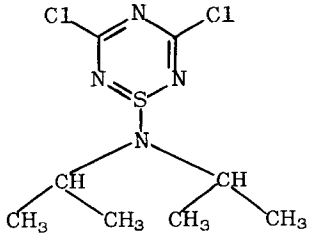

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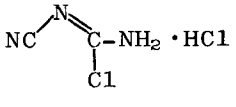

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The incorrect structure **1** was discussed[1] based on IR spectra with strong nitrile absorption. Schramm[2] showed that **2** in the KBr pellet prepared for IR spectroscopy easily hydrolyzes to N-cyano-chloroformamidine hydrochloride **4.**[4] The IR spectrum of **4** is identical with that expected for **1.**

The reaction of **2** with secondary amines, *e.g.* diisopropylamine leads to 1-diisopropylamino-3,5-dichloro-1λ⁴-2,4,6-thiatriazine **3**[2,3], CAS registry No. [54318-81-9].

Malodinitrile or sodium dicyanoamide reacts with phosphorus pentachloride[5] to analogous 2,2,4,6-tetrachloro-1,3,2λ⁵-diazaphosphorines or 2,2,4,6-tetrachloro-1,3,5,2λ⁵-triazaphosphorines.

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