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INTERESTING ERRORS IN SULFUR CHEMISTRY, 4

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CHLOROTHIO(DICHLOROFORMAMIDE)CYANAMIDE

In 1970 Geevers et al. 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%.

$$M^{\oplus} \begin{bmatrix} N & \text{CN} \\ N & \text{CN} \end{bmatrix}^{\ominus} \xrightarrow{\text{SOC1}_{2}, \text{ DMF}} C1-S-N \xrightarrow{\text{CN}} C2-N-C1$$

$$M^{\oplus} = \text{Na}; (CH_3)_4 N \qquad \qquad \underline{1}$$

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 ¹³C NMR, ESCA, IR, and crystal structure determination³ (compound 3) to possess the structure of 1,3,5-trichloro- $1\lambda^4$,2,4,6-thiatriazine 2.

The incorrect structure 1 was discussed¹ based on IR spectra with strong nitrile absorption. Schramm² 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. disopropylamine leads to 1-disopropylamino-3,5-dichloro- $1\lambda^4$ -2,4,6-thiatriazine $3^{2,3}$, CAS registry No. [54318-81-9].

Malodinitrile or sodium dicyanoamide reacts with phosphorus pentachloride⁵ to analogous 2,2,4,6-tetrachloro-1,3,2 λ^5 -diazaphosphorines or 2,2,4,6-tetrachloro-1,3,5, $2\lambda^5$ -triazaphosphorines.

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