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Journal of Sulfur Chemistry

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713926081>

Interesting Errors in Sulfur Chemistry, 1

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To cite this Article Senning, Alexander(1981) 'Interesting Errors in Sulfur Chemistry, 1', Journal of Sulfur Chemistry, 1: 6, 399

To link to this Article: DOI: 10.1080/01961778108082420

URL: <http://dx.doi.org/10.1080/01961778108082420>

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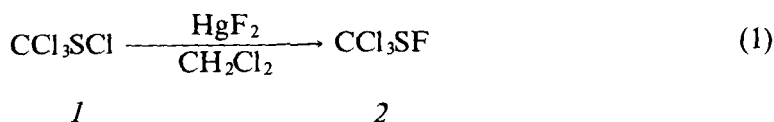
INTERESTING ERRORS IN SULFUR CHEMISTRY, 1

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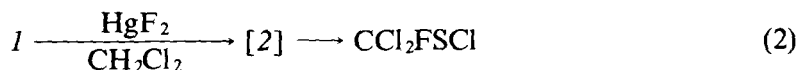
Trichloromethanesulfenyl Fluoride

In 1959 Kober¹ described the reaction of trichloromethanesulfenyl chloride **1** with mercury(II) fluoride and identified the reaction product as trichloromethanesulfenyl fluoride **2**:



Besides obtaining correct elemental analyses for his reaction product the author derivatized it with phthalimide. This derivative failed to depress the mixed melting point with authentic N-(trichloromethylthio)-phthalimide² (m.p. 177 °C).

Kober's claims were immediately disproved by Sheppard and Harris³ and by Kloosterziel⁴ who could show that the alleged **2** was in fact dichlorofluoromethanesulfenyl chloride **3**:



Later Kühle *et al.*⁶ demonstrated that authentic N-(dichlorofluoromethylthio)-phthalimide⁷ (m.p. 152–152.5 °C) does not depress the mixed melting point with N-(trichloromethylthio)-phthalimide.² According to Seel *et al.*⁸ the labile intermediate **2** can be isolated at low temperatures and characterized by ¹⁹F NMR.

In 1972 Kober's procedure for the synthesis of **3** was presented in a preparative organic chemistry handbook⁹ as a model preparation of a sulfenyl fluoride.

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