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Interesting Errors in Sulfur Chemistry, 10

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INTERESTING ERRORS IN SULFUR CHEMISTRY, 10

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METHYLATION OF TETRATHIOOXALATE¹

More than ten years ago Wawzonek and Heilmann² presumed 2,3,5,6-tetrakis(methyl-thio)-1,4-dithiin 1 as a side product of the electrochemical reduction of CS_2 , followed by methylation. The results were verified by Lodmell *et al.*³ by isolation of identical products.

Among other products a compound of supposed structure 1 was isolated from a complex reaction mixture, obtained by Jeroschewski and Hansen⁴ by methylation of tetramethylammonium tetrathiooxalate⁵.

Lund et al.⁶ could show that the methylation mixture of tetrathiooxalate contains a compound, identical with the compound isolated by Jeroschewski⁴ and confirmed by X-ray and ¹³C NMR as 4,5-bis(methylthio)-2-[bis(methylthio)methylene]-1,3-dithiole 2.

Previously 2 was obtained from dimethyl tetrathiooxalate and triphenylphosphine.

CH3S
$$C=C$$
 SCH3

4,5-bis(methylthio)-2-[bis(methylthio)-methylene]-1,3-dithiole CA Registry Number [78954-46-8]

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