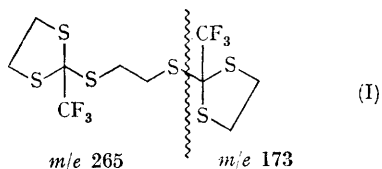


Condensation of Ethane-1,2-dithiol with Trifluoroacetic Acid

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REFLUXING trifluoroacetic acid affords an excellent medium for the preparation of thioketals and thioacetals, and is particularly effective with amino-aldehydes such as quinoline-4-carboxaldehyde. However, in the absence of other carbonyl compounds, ethane-1,2-dithiol undergoes an unprecedented condensation with the acid giving, in high yield, the orthothiolester (I).



When a solution of ethane-1,2-dithiol in an excess of trifluoroacetic acid was heated under reflux for 16 hr., compound (I) separated from

the (cooled) reaction mixture as colourless crystals, m.p. 85°, in 89% yield. Its structure derives from its spectroscopic properties [infrared bands at 1160, 1225, and 1280 cm^{-1} (C-F); n.m.r. singlets at 3.19 (4H) and 3.52 (8H) p.p.m.; mass spectrum shows intense peaks at m/e 173 and 265 but no M^+ peak], analysis and molecular weight.

Previous syntheses of ortho-thiol esters utilized reactive acid derivatives such as acyl chlorides^{1,2} and ortho-esters.³ Both formic and acetic acids condense with 4-methylbenzene-1,2-dithiol in the presence of perchloric acid but yield as products the corresponding dithiolium perchlorates.⁴

Other members of the class of compounds represented by compound (I) are being sought as potential hexadendate sulphur ligands.

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