

red within one second and is rapidly decomposed to black lead sulphide.

These experiments exclude structures of duplodithioacetone with one thioether bond and one trisulphide bond.

Further they point to a new method to obtain *gem* dithiols.

The author is indebted to Professor A. Fredga for his encouraging interest in this work.

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Studies on Peroxy Compounds XXII* Preparation of 2,3,4,5,4',5'- Hexahydro-2,2'-dithienyl

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2-Benzoyloxy tetrahydrothiophene, I, was first prepared in 1957 by Horner and Jürgens¹. The starting materials were benzoylperoxide and tetrahydrothiophene. Besides I, tetrahydrothiophene sulphoxide, benzoic acid and the anhydride of benzoic acid were obtained.

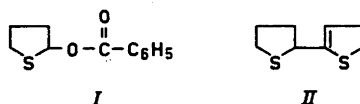
Sosnovsky^{2,3} prepared I by reacting tetrahydrothiophene with *t*-butyl perbenzoate in the presence of cuprous bromide. In that case no oxidation of the sulphide

* Part XXI. Lawesson, S.-O., Dahlén, M. and Frisell, C. *Acta Chem. Scand.* **16** (1962). *In press.*

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did occur. By pyrolysis of I for 2 h at 110°C a yield of 80 % of 2,3-dihydrothiophene was obtained. In another experiment Sosnovsky heated I in *t*-butanol under reflux for 100 h and isolated a quantitative yield of benzoic acid and a dimer of dihydrothiophene with a b.p. of 90°C/0.5 mm Hg, $n_D^{25} = 1.6006$. Mol. wt. 179. Nothing was said about the structure of the dimer. The present authors had at the same time⁴ performed this reaction. When we pyrolyzed I at 160–170°C for 2 h no 2,3-dihydrothiophene could be detected but a 45 % yield of the dimer was obtained. B.p. 88–90°C/0.2 mm Hg, $n_D^{20} = 1.6023$. Mol. wt. 168. Although 2,3-dihydrothiophene certainly is an intermediate in our case, the dimerization compound is the final product.



Gas chromatographic analysis showed one peak, indicating a single compound. I.R. measurements showed a double bond and elementary analyses agreed well with the formula, C₈H₁₂S₂. The dimer was desulphurated with Raney Ni and the hydrocarbon formed was octane which was shown by gas chromatographic analysis. The N.M.R. spectrum of the dimer was also taken and the relative intensities of the bands were 1:1:10, corresponding to one ethylenic, one tertiary and ten methylenic hydrogens. The configuration of the dimer was thus fixed to be II.

A full paper of these and related results is being prepared.

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