

SHORT
COMMUNICATIONS

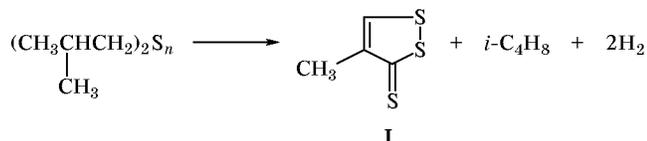
Unusual Thermolysis of Diisobutyl Polysulfides

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Gas-phase thermolysis of dialkyl polysulfides usually gives thiophene as the major product [1]. In the thermolysis of dibutyl polysulfides, a small amount (6%) of 5-methyl-1,2-dithiole-3-thione is also formed. We have found that the major product of thermolysis of a mixture of diisobutyl polysulfides ($(i\text{-C}_4\text{H}_9)_2\text{S}_n$ ($n = 3\text{--}4$) or each particular polysulfide at 350–400°C is 4-methyl-1,2-dithiole-3-thione (**I**). Its yield reaches 72% from diisobutyl trisulfide at 400°C. Also, 2-methylpropane-1-thiol (10–20%), diisobutyl sulfide (2–6%), and diisobutyl disulfide (2–17%) are obtained.



The scheme of formation of dithiolethione **I** implies intramolecular dehydrogenation of the alkyl

chain in polysulfides in the heterocyclization process which is thermodynamically favorable due to formation of pseudoaromatic system.

Thus, thermolysis of diisobutyl polysulfides is a convenient preparative method for the synthesis of 4-methyl-1,2-dithiole-3-thione (**I**).

Polysulfides were supplied in a stream of nitrogen into a quartz flow tubular reactor (0.65 × 0.03 m) which was heated in an electric furnace. Compound **I** was isolated by vacuum distillation of the liquid condensate. bp 144–146°C (3 mm), dark red liquid. ¹H NMR spectrum, δ, ppm: 2.21 d (CH₃), 8.10 d (CH, $J = 1$ Hz) (cf. [2]).

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2. Brown, R.F.C., Rae, I.D., and Sternhell, S., *Aust. J. Chem.*, 1965, vol. 18, p. 1211.