HIGH-TEMPERATURE SYNTHESIS OF 1,2-DITHIOL-3-ONE

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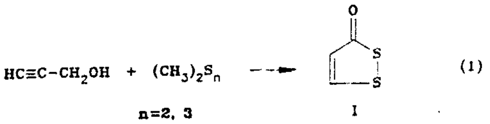
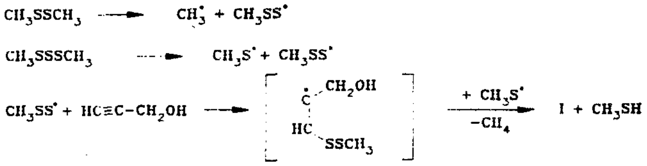
1,2-Dithiol-3-one (I) is, in general, prepared from the hard to obtain 1,2-dithiol-3-thione by a reaction with mercury acetate [1].

We found that compound I is the main liquid product in the gas phase reaction of propargyl alcohol with dimethyl di- or dimethyl trisulfide at 500°C:



At n = 2, its yield reaches 10%, and at n = 3, 25% (a 66...90% conversion of propargyl alcohol). Such low yields are due to an appreciable thermal decomposition of the starting compounds with the formation of gaseous thermolysis products. One to two percent of thiophene is formed together with compound I.

In reaction (1), propargyl alcohols probably acts as a trap for perthiyl radicals, generated during the thermal dissociation of di- or trisulfide:



A mixture of 74 g (~0.8 mole) of dimethyl trisulfide and 23.2 g (0.4 mole) of propargyl alcohol is fed in the course of 70 min in a nitrogen current (5 liter/h) into a tubular, hollow quartz reactor with a 650 × 30 mm heating zone. Thus 13.2 g of liquid condensate are obtained, containing 76% of thiolone I (yield 23%), 13% of the initial alcohol, and 2.5% of thiophene. Compound I is isolated by fractional distillation in vacuo (bp 111-115°C at 10 mm Hg) and purified by column chromatography in a 1:9 benzene–petroleum ether system. The spectral characteristics are identical to those given in [2].

LITERATURE CITED

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2. R. Mayer and J. Faust, Chem. Ber., 96, 2702 (1963).

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