24. Studies on the Syntheses of Ethylene Sulfide and Vinyl Thiol Acetate

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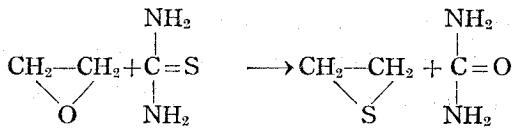
I. Synthesis of Ethylene Sulfide.

The authors have synthesized ethylene sulfide (I) with 50–60% yield by some improved method of G.P. 636708 according to the following reaction:



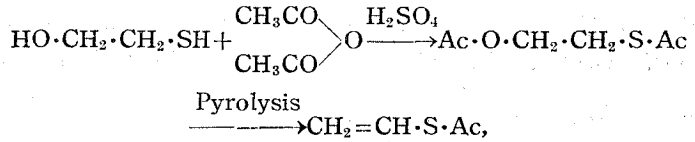
The chief improvements were as follows:

1) Ethyl alcohol was used as solvent.
2) Reaction temperature was maintained at 1–3°
3) Reaction mixture was neutralized by glacial acetic acid.

The polymerisation of (I) occurred easily.

The polymer was a white powder, m. 140–150° and insoluble in ordinary solvents.

II. Synthesis of Vinyl Thiol Acetate.



According to the above course, monothioethyleneglycol-diacetate, b. 98–99°/11mm, was prepared with 76% yield.

The diacetate was converted to vinyl thiol acetate (II), b. 121°, in 10–15% yield by passing the diacetate through a glass packed quarts tube heated to 500° in a CO_2 atmosphere.

In U.S.P. 2378535, it is stated that (II) can be polymerized by heating at 100° with 0.1% Bz_2O_2, or copolymerized with vinyl acetate to an infusible and insoluble resin. We could not however, obtain such a resin under the same conditions, but only a viscous liquid.

Further investigation on the chemical characters of ethylene sulfide is in progress.