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. $140-150^{\circ}$ and insoluble in ordinary solvents.

II. Synthesis of Vinyl Thiol Acetate.

$$\begin{array}{c} \text{HO} \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{SH} + \overset{\text{CH}_3 \text{CO}}{\overset{\text{CH}_2 \text{SO}_4}{\overset{\text{CH}_2 \text{SO}_4}{\overset{\text{CH}_2 \text{CO}}{\overset{\text{CH}_2 \cdot \text{CH}_2 \cdot \text{CH}_2}{\overset{\text{CH}_2 \cdot \text{CH}_2}{\overset{\text{CH}_2}{\overset{\text{CH}_2 \cdot \text{CH}_2}{\overset{\text{CH}_2}}{\overset{\text{CH}_2}{\overset{\text{CH}_2}{\overset{\text{CH}_2}}{\overset{\text{CH}_2}{\overset{\text{CH}_2}{\overset{\text{CH}_2}}{\overset{\text{CH}_2}{\overset{\text{CH}_2}}{\overset{\text{CH}_2}}{\overset{\text{CH}_2}}{\overset{\text{CH}_2}}{\overset{\text{CH}_2}}{\overset{\text{CH}_2}}{\overset{\text{CH}_2}{\overset{\text{CH}_2}}{\overset{\text{CH}_2}}{\overset{\text{CH}_2}}{\overset{\text{CH}_2}}{\overset{\text{CH}_2}}{\overset{\text{CH}_2}}{\overset{CH}_2}}{\overset{CH}_2}}}}}}}}}}}}}}}}}}}}}}}}}$$

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% Bz₂O₂, 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.