SYNTHESIS OF THIOPHENE FROM VINYL CHLORIDE

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It has been shown that thiophene is formed in 40% yield as a result of pyrolytic dehydrogenation of divinyl sulfide at 550°C [1].

We have found that a condensate consisting, according to the results of gas-liquid chromatography (GLC), of thiophene (60% yield based on the converted vinyl chloride, 40% conversion per pass) and thioacetaldehyde (30% yield based on the converted vinyl chloride) is formed when vinyl chloride (V = 4 liters/h) and hydrogen sulfide (V = 2 liters/h) are passed through an empty quartz tube heated to 550-560°.

$$\mathsf{CH}_2 \!\!=\! \mathsf{CHCI} \; + \; \mathsf{H}_2 \mathsf{S} \; \xrightarrow{-\mathsf{HCI}} \; \mathsf{CH}_2 \!\!=\! \mathsf{CHSH} \; \xrightarrow{+\mathsf{CH}_2 \!\!=\! \mathsf{CHCI}} \; \overset{\mathsf{CH}_2}{-\mathsf{HCI}} \; \overset{\mathsf{CH}_2}{\mathsf{CH}_2} \; \overset{\mathsf{CH}_2}{\mathsf{CH}_2} \; \xrightarrow{-\mathsf{H}_2} \; \overset{\mathsf{CH}_2}{\mathsf{CH}_2} \; \overset{\mathsf{CH}_2}{\mathsf{$$

The yield of thiophene is determined by the molar ratio of the reagents. The intermediate — ethenethiol — is partially isomerized to monomeric thioacetaldehyde, which is subsequently converted to the cyclic trimer. The primary reaction product is monomeric thioacetaldehyde when excess hydrogen sulfide is present.

The thiophene can be readily isolated from the condensate after treatment with aqueous alkali by rectification. Its physical constants and PMR and IR spectra are in complete agreement with the literature data.

The thioacetaldehyde was isolated in the form of the β -trithioacetaldehyde with mp 125°.

LITERATURE CITED

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