

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF IOWA STATE COLLEGE]

**REACTIONS IN LIQUID HYDROGEN SULFIDE. V.
REACTION WITH FURFURAL**

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Cahours¹ treated furfuralamide dissolved in alcohol with hydrogen sulfide gas and obtained a yellow crystalline substance which an analysis indicated was $C_{10}H_4S_2O_2$, thiofurfural. Baumann and Fromm² passed gaseous hydrogen sulfide into a solution of furfural dissolved in alcohol, saturated with hydrochloric acid, and isolated a similar body. Quam³ has shown that furfural is soluble in liquid hydrogen sulfide and reacts with it. An endeavor has been made to prepare the compound mentioned above by the reaction between the two liquids.

The furfural was added to the liquid hydrogen sulfide contained in glass tubes cooled in a bath of solid carbon dioxide and ether. These tubes were then sealed; on coming to room temperature a reaction started and after a few days the whole mass became semi-solid and some yellow crystals formed. The tubes were opened after a week, the excess of hydrogen sulfide was allowed to evaporate and any furfural unacted upon was dissolved out with ether. The residue was soluble in hot ethyl acetate, from which solution yellow monoclinic needles separated. These blackened easily in the air, the black material being insoluble in hydrogen sulfide. When boiled with dilute sodium hydroxide solution the material turned brown but this was soluble in ethyl acetate and the yellow crystals reappeared on evaporating. These crystals softened at 80° and melted between 95 and 98° , which agrees very well with the data for the polymerized material prepared by Baumann and Fromm which softened at 80° and melted from 90 – 92° . Two samples on analysis were found to contain 28.74 and 28.57% of sulfur, while the theoretical for the compound C_5H_4OS is 28.6%. This indicates that the compound is a mono-sulfur derivative of furfural.

Furan, furfural alcohol, pyromucic acid, the ethyl ester and the nitro-ethyl ester of pyromucic acid were all treated in a similar manner but no reaction occurred. Therefore, with furfural it is not the oxygen of the ring that is replaced but the one in the aldehyde group.

Other work in this Laboratory has shown that the aldehydes as a class react with liquid hydrogen sulfide and substitute a sulfur for an oxygen in the aldehyde group, thus liberating water. Since the water is not entirely miscible with liquid hydrogen sulfide, it appears in a separate layer in the tube.

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¹ Cahours, *Ann.*, **69**, 85 (1845).² Baumann and Fromm, *Ber.*, **24**, 3591 (1891).³ Quam, *THIS JOURNAL*, **47**, 103 (1925).