

L-ASCORBIC ACID IN CONCENTRATED SULFURIC ACID;
IMPROVED SYNTHESIS OF L-ASCORBIC ACID 6-SULFATE

by

DONALD W. LILLARD JR.

B. S., BOISE STATE UNIVERSITY, 1974

A MASTER'S THESIS

submitted in partial fulfillment of the
requirements for the degree

MASTER OF SCIENCE

Department of Grain Science and Industry

KANSAS STATE UNIVERSITY

Manhattan, Kansas

1977

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L-Ascorbic acid (I) does not dehydrate in concentrated sulfuric acid at room temperature, probably due to the formation of a hydroxyallyl cation structure. The c.m.r. spectrum of (I) dissolved in concentrated sulfuric acid showed that only two major components are present. The components were isolated by the following sequence. The reaction was quenched in cold ethyl ether ($\sim -65^{\circ}$), neutralized in the cold by addition of barium hydroxide, and the inorganic salts removed. The components were then separated and isolated using DEAE-cellulose ion-exchange column chromatography.

A total of four components were eluted from the column. The principal component (85%) was 6-sulfato-L-ascorbate (III), which was isolated in solid form as the barium salt in 48% yield. The other major component (5%) was thought to be 5-sulfato-L-ascorbate. The latter component was found to be unstable in base. When treated with alkali (pH 11.5 at 25°) the UV properties of the component changed, indicating elimination of the sulfate group to form a product thought to be 2,3,4,6-tetrahydroxy-2,4-hexadienoate- γ -lactone. This last compound was thought to be one of the two remaining minor components eluted from the column. The fourth component remains unidentified.

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