Furfural as a new decomposition product of glucose solution under oxygen atmosphere

HIROYASU OGATA*, TOSHIO SHIBAZAKI, AKIRA EJIMA, TOSHIO MATSUURA, FUMIKO SHIMADA, *Division of Drug, National Institute of Hygienic Sciences, Setagaya-ku, Tokyo, Japan*

The decomposition of glucose under autoclaving and storage is one of the major problems in the quality control of parenteral medication. It is well known that glucose is converted to 5-hydroxymethyl-2-furfural (5-HMF), thence to formic acid and levulinic acid (Heimlich & Martin, 1960). Although it had also been suggested (Taylor, Jappy & Neil, 1971) that a component intermediate between glucose and 5-HMF is responsible for the decrease in pH observed during autoclaving, the general assumption is that this decrease in pH arises rather from organic acids formed by further decomposition of 5-HMF itself. The mechanism of glucose decomposition is far from clear.

By h.p.l.c. analysis of the decomposition products of glucose in aqueous solution, furfural has now been identified. Two ml of 1·0% glucose solution was sealed under oxygen or nitrogen as control in 10 ml volume ampoules. After heating at 100° for 5 days, 10 μl of each solution was analysed by h.p.l.c. From those solutions sealed under oxygen two components were separated from unchanged glucose. These had retention volumes of 10·0 and 17·4 ml. This glucose solution was extracted with diethyl ether, and the organic layer was washed with 0·1M NaOH then with water. After evaporation to a small volume under nitrogen two well resolved peaks were obtained on gas chromatography, 5-HMF (Rt 17 min) and furfural (Rt 4 min). The latter was identified from its spectrum (Fig. 1). Its R_F value (t.l.c.), retention time (h.p.l.c. and g.c.), its ultraviolet spectrum in addition, were all identical with those of an authentic sample. 5-HMF was also compared with an authentic sample.

Furfural was formed from glucose or from 5-HMF when heated in the presence of oxygen particularly in solutions of acid pH. This is the first report that glucose (hexose) oxidatively decomposes to furfural in aqueous solution, though it is well known that the pentose in acidic solution decomposes in this way, and that furfural is formed as a pyrolytic degradation product of glucose (Houminer & Patai, 1969; Kato, 1969).

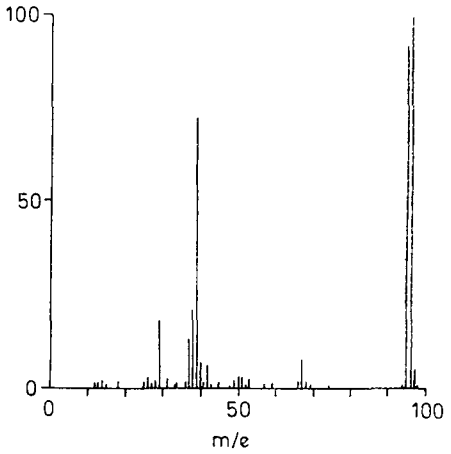

FIG. 1. Mass spectrum of unknown decomposition product (X). Mass spectrum was measured by g.c.-m.s., LKB-9000 (Shimazu). G.c. conditions: 5% diethylene glycol succinate polyester on Chromosorb W (60–80 mesh), 3 mm × 1 m, column temp.; 50°, ms condition: ion source temp.; 290°, electron energy; 70 eV, trap curr.; 60 μA, accelerated volt.; 3·5 kV.

* Correspondence.

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