

## Gas-liquid chromatographic assay of the components of electrochemically reduced glucose solutions

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**Introduction.** No method could be found in the literature for the simultaneous determination of D-glucose and its derivatives obtained either by catalytic hydrogenation or by electrolysis. The production of sorbitol by an electrochemical process (used on an industrial scale before the second world war) has been re-examined recently owing to its several advantages in comparison with catalytic hydrogenation procedures. Owing to the very similar characteristics of the byproducts obtained in the reduction processes, such as deoxy sugars and/or deoxy alditols, the analytical methods used proved to be unsatisfactory in both qualitative and quantitative analysis. Our previously described gas-liquid chromatographic (GLC) method has therefore been applied, after optimization of the conditions, for the simultaneous determination of D-glucose, 2-deoxy-D-glucose, D-glucitol and 2-deoxy-D-glucitol.

**Materials and methods.** *D-Glucose* and 2-deoxy-D-glucose were purchased from Serva (Heidelberg, F.R.G.). Sorbitol samples 1-6 were products of various companies as follows: Peti Nitrogen Metivek, Pet, Hungary (sorbitol 1), Atlas Powder, Miami, FL, U.S.A. (sorbitol 2) Anil Starch Products, Ahmedabad, India (sorbitol 3) SPA, Milan, Italy (sorbitol 4), an unknown Japanese company (sorbitol 5) and Carlo Erba, Milan, Italy (sorbitol 6). 2-Deoxy-D-glucitol was prepared from 2-deoxy-D-glucose by reduction with sodium tetrahydroborate as described by Lehrfeld. Test samples 2-7 were electrochemically reduced glucose solutions (1 A/4) containing sodium sulphate (0.5 A/4) or, in test sample 5- lithium nitrate (0.5 A/4). The electrochemical reductions were carried out in an Electra MP Cell (500 cm<sup>3</sup>) equipped with an ion-exchange membrane (Nafion XR 423) and separated cathode and anode compartments (Electra Cell, Taby, Sweden). Electrolyses were performed with current densities of 8 A per 200 cm<sup>3</sup> for 6 h (test sample 2) and of 4 A per 200 cm<sup>3</sup> for 6.7h (3-5) for 4 h (6) and for 4 x 6.7 h (7). Test sample 1, an unelectrolyzed solution of D-glucose, served as the basis of comparison.

**Results and discussion.** Our model investigations, performed with authentic saccharides in order to find the optimum derivatization and GLC conditions, gave the following results: Columns 0.5, 2 and 3 m long (each of 4 mm I.D.) coated with 15% Dexsil GC 300 or 3% SP 2250 were tested in parallel. [Columns 0.5 m long served only for testing saccharides of higher degree of polymerization (DP); in the present analysis the proportion of saccharides and/or alditols with DP > 1 was <0.1 wt.-% in all the samples tested.] Optimum separation was achieved on the 15% Dexsil columns (3 m x 4 mm I.D.) with the sugars as their TMS oxime derivatives.

**Conclusion.** The differences in time of retention of the components are summarized and simplified by the chromatogram as a single peak which is obtained for the 2-deoxy-D-glucose derivative.