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Note

Preparation of macro quantities of glucose oligomers

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Glucose and its oligomers are often required as standards in the gas–liquid and high-performance liquid chromatographic analysis of glucose syrups. Although glucose and maltose are freely available, the maltose often contains significant amounts of glucose and maltotriose. The higher oligomers (up to maltohexaose) may also be obtained commercially but their cost is often prohibitive and their purity suspect. We have developed a method whereby macro quantities of glucose oligomers from maltotriose to maltopentaose may be produced by fractionation of the acetate esters of the glucose syrup. The method can easily be extended to include the separation of higher saccharides if so desired.

METHOD AND RESULTS

A 10-g amount of 43 D.E. glucose syrup solids were dried at 80° under reduced pressure for 24 h and then transferred to a 500-ml flask. Volumes of 100 ml of dried pyridine and 50 ml acetic anhydride were added and the contents of the flask stirred at 60° using a calcium chloride filter to exclude moisture. The treatment was continued for 1 h after the solids had dissolved to ensure complete acetylation. After this time the contents were poured into 1 l of crushed ice and water. The precipitated white solid was filtered off, dissolved in chloroform and washed with 10% sulphuric acid, saturated sodium bicarbonate and water respectively and dried (sodium sulphate). The chloroform extract was finally dried onto a portion of the column material (silica gel, Kieselgel 60, 70–230 mesh) and was separated on a 100 × 3 cm I.D. glass column using benzene–ethyl acetate (2:1) as solvent. The flow-rate was approximately 400 ml/h and an automatic fraction collector was used to collect the effluent in 10-ml units. The contents of every tenth tube were chromatographed on silica gel thin-layer plates (benzene–ethyl acetate (3:1) as solvent) and the results of this examination are shown in Table I. After fraction number 250 had been collected the solvent rate was decreased (benzene–ethyl acetate (3.2)) to increase the elution rate.

The glucose oligomers were deacetylated by stirring with 0.1 *N* sodium methoxide, demineralized using Zerolit DM-F mixed ion-exchange resin (CO_3^{2-} form) and dried to syrups by evaporation under reduced pressure.

The glucose syrup solids used as starting material may be changed according

TABLE I

ANALYSIS OF EFFLUENT FROM COLUMN

Fraction number (10-ml fractions)	*Carbohydrate composition*	*Volume (ml)*	*Approximate carbohydrate content of fractions (mg/ml)*
0–50	0	500	0
50–70	Glucose	200	12
70–100	Glucose and maltose	300	6
100–150	Maltose	500	4
150–180	Maltose and maltotriose	300	2
180–250	Maltotriose	700	2
250–270	Maltotriose	200	
270–290	Maltotriose and maltotetraose	200	3
290–360	Maltotetraose	700	2
360–450	Maltotetraose and maltopentaose	900	1
450–500	Maltopentaose	500	2

to the oligomers of interest in the separation. Total separation time in this experiment was 14 h but optimization of the conditions of separation would undoubtedly reduce this.

Although only acetate esters were separated in this instance, benzoate esters should also prove as useful if not more so than acetates owing to the larger differences in molecular weight between oligomers.