INTERNATIONAL STANDARD

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Starch derivatives — Determination of the composition of glucose syrups, fructose syrups and hydrogenated glucose syrups — Method using high-performance liquid chromatography

Produits dérivés de l'amidon — Détermination de la composition des sirops de glucose, des sirops de fructose et des sirops de glucose hydrogénés — Méthode par chromatographie en phase liquide à haute performance

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2. www.iso.org/directives

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

The committee responsible for this document is ISO/TC 93, *Starch (including derivatives and by-products)*.

This second edition cancels and replaces ISO 10504:1998, of which it constitutes a minor revision.

Starch derivatives — Determination of the composition of glucose syrups, fructose syrups and hydrogenated glucose syrups — Method using high-performance liquid chromatography

1 Scope

This International Standard describes a high-performance liquid chromatographic (HPLC) method for measuring the composition of dextrose solutions, glucose syrups, fructose-containing syrups, hydrogenated glucose syrups, sorbitol, mannitol and maltitol. The constituents are mainly glucose, maltose, maltotriose, fructose, sorbitol, mannitol, maltitol and malto-oligosaccharides.

The use of a column packed with cation-exchange resin is essential.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 5381:1983, *Starch hydrolysis products — Determination of water content — Modified Karl Fischer method*

3 Principle

Saccharide components are separated using high-performance liquid chromatography. Separation is achieved using a cation-exchange column with water as the eluent. The eluted components are detected by means of a differential refractometer, and quantified using an electronic integrator.

4 Reagents

All reagents used shall be of recognized analytical reagent grade.

4.1 Special distilled water.

The water used may be double-distilled of quality grade 1 in accordance with ISO 3696. The most suitable is demineralized water, which prevents contamination of the ion-exchange resin.

The water should be filtered by passage through a 0,22 µm filter. Also, it should be degassed by treatment under vacuum, or by use of an in-line degassing unit. The water should be maintained under an inert atmosphere, and preferably at 70 °C to inhibit microbial growth.

NOTE Some commercial water-purification devices produce water which is both filtered and degassed.

4.2 Primary standard solutions.

Prepare solutions (see Annex A) containing 10 % (or less) dry matter, according to the sensitivity of the refractometer, with compositions as close as possible to that of the samples to be analysed.

NOTE Suitable reference materials for the constituents listed in Clause 1 can be obtained from established chemical companies.

4.3 Ion-exchange resins, for off-line demineralization of samples.

Salts present in the sample will co-elute from the column, and will be detected by the refractometer, causing errors in the determination. These salts shall first be removed by ion-exchange resins. The most convenient way is to have an in-line guard column cartridge system (5.5), but this may also be carried out off-line using the following resins[1)]:

a) Cation type:

   1) strong cation exchanger, 4 % cross-linked polystyrene divinylbenzene, in the H^+ form;

   2) 200 mesh to 400 mesh in the dry form;

b) Anion type:

   1) weak anion exchanger, 4 % cross-linked polystyrene divinylbenzene support containing tertiary amine groups, in the free base form;

   2) 200 mesh to 400 mesh in the dry form.

5 Apparatus

5.1 Liquid chromatograph; equipped with the following.

5.1.1 Pump, pulseless, that delivers a constant flow, at the rate required.

5.1.2 Differential refractometer, thermostatically controlled.

5.1.3 Thermostatically controlled column oven, capable of maintaining the column at temperatures up to 95 °C, to within ± 0,5 °C.

5.2 Sample injector, comprising a loop injector (manual or part of autosampler) with a capacity of 20 µl or less.

5.3 Integrator, comprising an electronic integrator with calculating and recording capabilities, compatible with the voltage output of the detector.

5.4 Separation column, comprising a pre-packed cation-exchange column in the form best suited for the analysis. The recommended resin is 6 % to 8 % cross-linked sulfonated polystyrene divinylbenzene with a bead diameter of 9 µm to 25 µm.

NOTE Acceptable columns are available from several major column suppliers.

5.5 Guard columns, custom-prepared dual-cartridge system, inserted unheated in-line, to demineralize the sample.[2)]

5.6 Sample filtration system, comprising a syringe to which suitable membrane disc filters can be attached. These should be of 0,45 µm pore size.

Commercially available syrups are usually highly refined, and a 0,45 µm filter is suitable. However, if blockage of the chromatograph is too frequent, a 0,22 µm filter should be used.

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1) While resins meeting these specifications are available from more than one supplier, their performance is variable. Experience in several laboratories has shown that the resins AG® 50W-X4 and AG® 3-X4 perform satisfactorily. (AG® 50W-X4 and AG® 3-X4 are trade names of products supplied by Bio-Rad. This information is given for the convenience of the users of this International Standard and does not constitute an endorsement by ISO of these products. Equivalent products may be used if they can be shown to lead to the same results.)

2) There are a few systems available but with varying efficiency. The Bio-Rad guard cartridges 125–0118 have been shown in several laboratories to be the most effective in all respects. (This information is given for the convenience of the users of this International Standard and does not constitute an endorsement by ISO of these products. Equivalent products may be used if they can be shown to lead to the same results.)