

Foodstuffs — Determination of isomalt, lactitol, maltitol, mannitol, sorbitol and xylitol in foodstuffs

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National foreword

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Produits alimentaires - Dosage de l'isomalt, du lactitol, du maltitol, du mannitol, du sorbitol et du xylitol dans les produits alimentaires

Lebensmittel - Bestimmung von Isomalt, Lactit, Maltit, Mannit, Sorbit und Xylit in Lebensmitteln

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Foreword

This European Standard (EN 15086:2006) has been prepared by Technical Committee CEN/TC 275 "Food analysis - Horizontal methods", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2006, and conflicting national standards shall be withdrawn at the latest by September 2006.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This European Standard specifies an HPLC-method for the determination of isomalt and other polyols such as lactitol, maltitol, mannitol, sorbitol and xylitol in foodstuffs. Chemically isomalt is described as a mixture of 6-O- α -D-glucopyranosyl-D-sorbitol (1,6-GPS) and 1-O- α -D-glucopyranosyl-D-mannitol (1,1-GPM).

The method has been validated in a collaborative study for isomalt (sum of GPS and GPM) on cookies, chewing gum, chocolate and on hard candies. Validation data for GPS and GPM are given in Clause 8, Annex A, Tables A.1 and A.2

The determination of the other sugar alcohols has been validated in a further collaborative study using the same method. The samples were pudding (lactitol, mannitol, xylitol), cookies (lactitol, maltitol, mannitol, sorbitol and xylitol), hard candies (lactitol, mannitol, xylitol, sorbitol) and chewing gum (maltitol, mannitol, sorbitol). Validation data are given in Clause 8 and Annex A, Tables A.3 to A.7.

2 Normative references

The following referenced documents are indispensable for the application of this European Standard. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)*

3 Principle

The sample is diluted, dissolved or extracted with water and possibly filtered. If necessary the sample is clarified using modified Carrez solutions. The polyols are separated by HPLC on cation exchanger with Ca^{++} or Pb^{++} counter ion using high purity water at 60 °C to 80 °C, detected using a refractive index detector (differential refractometer, RI-detector), and determined by the external standard method [1].

4 Reagents

4.1 General

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and water of at least grade 1 according to EN ISO 3696 or use distilled water.

4.2 Standard substances

4.2.1 General

When using standard substances containing constitutional water, this content has to be taken into account.

Example: molar mass of GPS = 344,32 g/mol, GPM dihydrate = 380,32 g/mol. The exact water contents of the standard substances are determined by Karl-Fischer titration.

Alternative to the calibration with pure substances, an isomalt reference sample with exactly known mass concentrations of GPM and GPS can be used.

4.2.2 6-O- α -D-Glucopyranosyl-D-sorbitol (1,6 GPS), without constitutional water.

4.2.3 1-O- α -D-Glucopyranosyl-D-mannitol (1,1-GPM)¹⁾, crystallizes with 2 mole of water (water content approximately 10 %).

4.2.4 Lactitol, crystallizes with 1 mole of water (water content approximately 5 %).

4.2.5 Maltitol

4.2.6 Mannitol

4.2.7 Sorbitol

4.2.8 Xylitol

4.3 Standard solutions

Dissolve appropriate amounts of polyol standard substances (4.2.2 to 4.2.8) in water and dilute this solution again with water to obtain standard solutions with a total mass concentration of approximately 2 g/100 ml for the sum of all components.

This solution may be stored for 6 weeks in a refrigerator set at +4 °C. Alternatively it is possible to store the standard solution deep frozen (-18 °C) for up to 1 year.

4.4 Carrez solution I, modified

Dissolve 53,45 g of potassium hexacyanoferrate(II) ($K_4[Fe(CN)_6] \cdot 3 H_2O$) in water, mix well and dilute to 500 ml with water. Store in a brown bottle and replace it regularly.

4.5 Carrez solution II, modified

Dissolve 148,75 g of zinc nitrate ($Zn(NO_3)_2 \cdot 6 H_2O$) in water, mix well and dilute to 500 ml with water. Store in a brown bottle and replace it regularly.

5 Apparatus

5.1 General

Usual laboratory apparatus and, in particular, the following:

5.2 Membrane filter, for filtering the sample test solution, with pore size of e.g. 0,45 μm .

NOTE Filtering of the mobile phase as well as of the sample solution through a membrane filter prior to use or injection is supposed to increase longevity of the columns.

¹⁾ For the availability of the standard substance, contact your National Standardization Institute.

5.3 Magnetic stirrer

5.4 HPLC system, consisting of a pump, a sample injecting device, a refractive index (RI) detector with variable temperature setting, a column oven and an evaluating system.

5.5 Analytical separation column, e.g. 300 mm x 7,8 mm, filled with ion exchange resin with Ca^{++} or Pb^{++} counter ion. To protect the analytical column, it is recommended to use a pre-column of similar characteristics. Shorter columns (e.g. 100 mm x 7,8 mm) may cause insufficient separations.

6 Procedure

6.1 Preparation of the test sample

Homogenize the test sample. This can be achieved by grinding solid samples as hard candies or chocolate with an appropriate mill at low temperatures, and homogenizing subsequently by mixing the ground sample. Deep freeze chewing gum before grinding. Homogenize semi solid samples e.g. ice cream by melting and stirring.

Treat turbid solutions which can occur when preparing e.g. cakes and pastries with modified Carrez solutions.

NOTE Although it has not been tested in the inter-laboratory study, it is recommended to de-ionize sample test solutions, if treated with Carrez solutions in order to protect the separation column. Ions introduced by these reagents may be eliminated by means of appropriate ion exchange resins (e.g. de-ashing systems, cartridges) prior to the injection on the analytical column.

6.2 Preparation of the sample test solution

6.2.1 Soluble samples (e.g. hard candies, compressed products)

Weigh in 2 g of the homogenized sample in a 100 ml volumetric flask and dissolve with water, mix and dilute to the mark. Filter turbid solutions through a membrane filter (5.2) or clarify with modified Carrez solutions (4.4) (4.5).

6.2.2 Incompletely soluble samples (e.g. pastries, chewing gum, chocolate)

Weigh in 5 g to 10 g of the homogenized sample in a 100 ml volumetric flask and mix with 50 ml of water, stir for 30 min at 40 °C to 60 °C with a magnetic stirrer and clarify with modified Carrez solutions. Let the flask stand to reach room temperature and dilute to the mark with water. When the precipitate has settled, filter the supernatant aqueous phase through a membrane filter (5.2). Fat containing sample solutions may sometimes need to be filtered a second time through a membrane filter of smaller pore size.