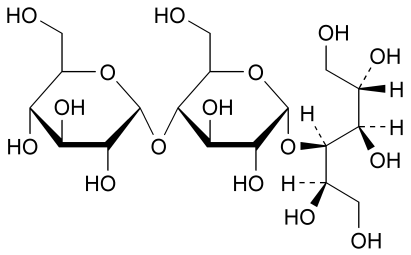
IMPURITIES

A. sorbitol,



B. *O*-α-D-glucopyranosyl-(1→4)-*O*-α-D-glucopyranosyl-(1→4)-D-glucitol (maltotriitol).

01/2008:1236

MALTITOL, LIQUID

Maltitolum liquidum

DEFINITION

Aqueous solution of a hydrogenated, partly hydrolysed starch, composed of a mixture of mainly 4-*O*-α-D-glucopyranosyl-D-glucitol (D-maltitol) with D-glucitol (D-sorbitol) and hydrogenated oligo- and polysaccharides.

Content:

– *D-maltitol* ($C_{12}H_{24}O_{11}$): minimum 50.0 per cent *m/m* (anhydrous substance) and 95.0 per cent to 105.0 per cent of the content stated on the label;
– *D-sorbitol* ($C_6H_{14}O_6$): maximum 8.0 per cent *m/m* (anhydrous substance);
– *anhydrous substance*: 68.0 per cent *m/m* to 85.0 per cent *m/m*.

CHARACTERS

Appearance: clear, colourless, syrupy liquid.

Solubility: miscible with water and with glycerol.

IDENTIFICATION

First identification: A.

Second identification: B, C.

A. Examine the chromatograms obtained in the assay.

Results: the principal peak in the chromatogram obtained with the test solution is similar in retention time to the principal peak in the chromatogram obtained with reference solution (a).

B. Thin-layer chromatography (*2.2.27*).

Test solution. Dilute 0.35 g of the substance to be examined to 100 ml with *water R*.

Reference solution (a). Dissolve 20 mg of *maltitol CRS* in *water R* and dilute to 10 ml with the same solvent.

Reference solution (b). Dissolve 20 mg of *maltitol CRS* and 20 mg of *sorbitol CRS* in *water R* and dilute to 10 ml with the same solvent.

Plate: *TLC silica gel G plate R*.

Mobile phase: *water R, ethyl acetate R, propanol R* (10:20:70 *V/V/V*)

Application: 2 µl.

Development: over a path of 17 cm.

Drying: in air.

Detection: spray with *4-aminobenzoic acid solution R*. Dry in a current of cold air until the acetone is removed. Heat at 100-105 °C for 15 min. Allow to cool and spray with a 2 g/l solution of *sodium periodate R*. Dry in a current of cold air. Heat at 100 °C for 15 min.

System suitability: reference solution (b):

– the chromatogram shows 2 clearly separated spots.

Results: the principal spot in the chromatogram obtained with the test solution is similar in position and colour to the principal spot in the chromatogram obtained with reference solution (a).

C. To 3 ml of a freshly prepared 100 g/l solution of *pyrocatechol R*, add 6 ml of *sulphuric acid R* while cooling in iced water. To 3 ml of the cooled mixture, add 0.3 ml of solution S (see Tests). Heat gently over a naked-flame for about 30 s. A pink colour develops.

TESTS

Solution S. Dilute 7.0 g to 50 ml with *water R*.

Appearance of solution. Solution S is clear (*2.2.1*) and colourless (*2.2.2, Method II*).

Conductivity (*2.2.38*): maximum 10 µS·cm^{-1}, measured on undiluted liquid maltitol while gently stirring with a magnetic stirrer.

Reducing sugars: maximum 0.2 per cent, calculated as glucose equivalent.

To 5.0 g add 6 ml of *water R*, 20 ml of *cupri-citric solution R* and a few glass beads. Heat so that boiling begins after 4 min and maintain boiling for 3 min. Cool rapidly and add 100 ml of a 2.4 per cent *V/V* solution of *glacial acetic acid R* and 20.0 ml of *0.025 M iodine*. With continuous shaking, add 25 ml of a mixture of 6 volumes of *hydrochloric acid R* and 94 volumes of *water R* and, when the precipitate has dissolved, titrate the excess of iodine with *0.05 M sodium thiosulphate* using 1 ml of *starch solution R*, added towards the end of the titration, as indicator. Not less than 12.8 ml of *0.05 M sodium thiosulphate* is required.

Lead (*2.4.10*): maximum 0.5 ppm.

Nickel (*2.4.15*): maximum 1 ppm.

Water (*2.5.12*): 15.0 per cent *m/m* to 32.0 per cent *m/m*, determined on 0.100 g. Use as solvent a mixture of equal volumes of *anhydrous methanol R* and *formamide R*. Carry out the titration at about 50 °C.

ASSAY

Liquid chromatography (*2.2.29*).

Test solution. Mix 1.00 g of the solution to be examined with 20 ml of *water R* and dilute to 50.0 ml with the same solvent.

Reference solution (a). Dissolve 50.0 mg of *maltitol CRS* in 2 ml of *water R* and dilute to 5.0 ml with the same solvent.

Reference solution (b). Dissolve 8.0 mg of *sorbitol CRS* in 2 ml of *water R* and dilute to 5.0 ml with the same solvent.

Reference solution (c). Dissolve 50 mg of *maltitol R* and 50 mg of *sorbitol R* in 2 ml of *water R* and dilute to 5.0 ml with the same solvent.

Column:

– *size*: *l* = 0.3 m, Ø = 7.8 mm;
– *stationary phase*: *strong cation exchange resin (calcium form) R* (9 µm);
– *temperature*: 85 ± 2 °C.

Mobile phase: degassed *water R*.

Flow rate: 0.5 ml/min.

Detection: refractometer maintained at a constant temperature.

Injection: 20 µl.

Run time: 3 times the retention time of maltitol.

Relative retention with reference to maltitol (retention time = about 16 min): sorbitol = about 1.8.

System suitability: reference solution (c):

– *resolution*: minimum 2 between the peaks due to sorbitol and maltitol.

Calculate the percentage contents of D-maltitol and D-sorbitol from the declared contents of *maltitol CRS* and *sorbitol CRS*.

LABELLING

The label states the content of D-maltitol.

01/2008:1542
corrected 6.0

MALTODEXTRIN

Maltodextrinum

DEFINITION

Mixture of glucose, disaccharides and polysaccharides, obtained by the partial hydrolysis of starch.

The degree of hydrolysis, expressed as dextrose equivalent (DE), is not greater than 20 (nominal value).

CHARACTERS

Appearance: white or almost white, slightly hygroscopic powder or granules.

Solubility: freely soluble in water.

IDENTIFICATION

A. Dissolve 0.1 g in 2.5 ml of *water R* and heat with 2.5 ml of *cupri-tartaric solution R*. A red precipitate is formed.

B. Dip, for 1 s, a suitable stick with a reactive pad containing glucose-oxidase, peroxidase and a hydrogen-donating substance, such as tetramethylbenzidine, in a 100 g/l solution of the substance to be examined. Observe the colour of the reactive pad; within 60 s the colour changes from yellow to green or blue.

C. It is a powder or granules.

D. Dextrose equivalent (see Tests).

TESTS

Solution S. Dissolve 12.5 g in *carbon dioxide-free water R* and dilute to 50.0 ml with the same solvent.

pH (*2.2.3*): 4.0 to 7.0.

Mix 1 ml of a 223.6 g/l solution of *potassium chloride R* and 30 ml of solution S.

Sulphur dioxide (*2.5.29*): maximum 20 ppm.

Heavy metals (*2.4.8*): maximum 10 ppm.

Dilute 4 ml of solution S to 30 ml with *water R*. The solution complies with test E. Prepare the reference solution using 10 ml of *lead standard solution (1 ppm Pb) R*.

Loss on drying (*2.2.32*): maximum 6.0 per cent, determined on 10.00 g by drying in an oven at 105 °C.

Sulphated ash (*2.4.14*): maximum 0.5 per cent, determined on 1.0 g.

Dextrose equivalent (DE): within 2 DE units of the nominal value.

Weigh an amount of the substance to be examined equivalent to 2.85-3.15 g of reducing carbohydrates, calculated as dextrose equivalent, into a 500 ml volumetric flask. Dissolve in *water R* and dilute to 500.0 ml with the same solvent. Transfer the solution to a 50 ml burette.

Pipette 25.0 ml of *cupri-tartaric solution R* into a 250 ml flask and add 18.5 ml of the test solution from the burette, mix and add a few glass beads. Place the flask on a hot plate, previously adjusted so that the solution begins to boil within 2 min ± 15 s. Allow to boil for exactly 120 s, add 1 ml of a 1 g/l solution of *methylene blue R* and titrate with the test solution (V_1) until the blue colour disappears. Maintain the solution at boiling throughout the titration.

Standardise the cupri-tartaric solution using a 6.00 g/l solution of *glucose R* (V_0).

Calculate the dextrose equivalent using the following expression:

$$\frac{300 \times V_0 \times 100}{V_1 \times M \times D}$$

V_0 = total volume of glucose standard solution, in millilitres,

V_1 = total volume of test solution, in millilitres,

M = sample mass, in grams,

D = percentage content of dry matter in the substance.

Microbial contamination. Total viable aerobic count (*2.6.12*) not more than 10^3 bacteria and 10^2 fungi per gram, determined by plate count. It complies with the tests for *Escherichia coli* and *Salmonella* (*2.6.13*).

LABELLING

The label states the dextrose equivalent (DE) (= nominal value).

01/2008:2355

MANDARIN OIL

Citri reticulatae aetheroleum

DEFINITION

Essential oil obtained without heating, by suitable mechanical treatment, from the peel of the fresh fruit of *Citrus reticulata* Blanco.

CHARACTERS

Appearance: greenish, or yellow to reddish orange liquid showing blue fluorescence.

It has a characteristic odour.

IDENTIFICATION

First identification: B.

Second identification: A.

A. Thin-layer chromatography (*2.2.27*).

Test solution. Dilute 0.1 ml of the substance to be examined to 1 ml with *toluene R*.

Reference solution. Dissolve 2 µl of *methyl N-methylanthranilate R*, 4 mg of *guaiazulene R* and 10 mg of *α-terpineol R* in 10 ml of *toluene R*.

Plate: *TLC silica gel plate R* (5-40 µm) [or *TLC silica gel plate R* (2-10 µm)].

Mobile phase: *ethyl acetate R, toluene R* (15:85 *V/V*).

Application: 10 µl [or 2 µl] as bands.

Development: over a path of 15 cm [or 6 cm].

Drying: in air.