Edition: BP 2025 (Ph. Eur. 11.6 update)

Syrup

General Notices

(Liquid Sucrose, Ph. Eur. monograph 2797)

Ph Eur

DEFINITION

Aqueous solution of sucrose.

Content

— *dry matter*: 66.0 per cent *m/m* to 67.5 per cent *m/m*.

CHARACTERS

Appearance

Clear, colourless or pale yellow, viscous liquid.

Solubility

Miscible with glycerol.

IDENTIFICATION

A. Liquid chromatography (2.2.29) as described in the test for related substances with the following modification.

Injection Test solution (b) and reference solution (c).

Results The principal peak in the chromatogram obtained with test solution (b) is similar in retention time and size to the principal peak in the chromatogram obtained with reference solution (c).

B. Dilute 75 g to 100 mL with *water R*. Dilute 1 mL of the solution to 100 mL with *water R*. To 5 mL of this solution add 0.15 mL of freshly prepared *copper sulfate solution R* and 2 mL of freshly prepared *dilute sodium hydroxide solution R*. The solution is blue and clear and remains so after boiling. To the hot solution add 4 mL of *dilute hydrochloric acid R* and boil for 1 min. Add 4 mL of *dilute sodium hydroxide solution R*. An orange precipitate is formed immediately.

TESTS

Colour value

Maximum 30.

Dilute a quantity of the substance to be examined corresponding to 50.0 g of dry matter (see Tests) to 100.0 g with *water R*. Filter through a membrane filter (nominal pore size 0.45 µm) and allow to stand for about 15 min. Measure the absorbance (*2.2.25*) at 420 nm, using a minimum path length of 5 cm (a path length of 10 cm or more is preferred).

Calculate the colour value using the following expression:

$$\frac{A}{b \cdot c}$$

A	=	absorbance measured at 420 nm;
b	=	path length, in centimetres;
c	=	concentration of the solution obtained, in grams per millilitre, calculated from the refractive index (*2.2.6*) of the solution; use Table 2797.-1 and interpolate the values if necessary.

Table 2797.-1

	***c* (g/mL)**
1.4138	0.570
1.4159	0.585
1.4179	0.600
1.4200	0.615
1.4221	0.630
1.4243	0.645
1.4264	0.661

System suitability:

— *repeatability*: the absolute difference between 2 results is not greater than 3.

Conductivity (*2.2.38*)

Maximum 35 µS·cm^{-1} at 20 °C.

Dilute a quantity of the substance to be examined corresponding to 31.3 ± 0.1 g of dry matter (see Tests) to 100.0 mL with *carbon dioxide-free water R*. Measure the conductivity of the solution (C_1), while gently stirring with a magnetic stirrer, and that of the water used for preparing the solution (C_2). The readings must be stable within 1 per cent over a period of 30 s. Calculate the conductivity of the solution of the substance to be examined using the following expression:

Related substances

Liquid chromatography (*2.2.29*).

Test solution (a) Dilute 0.200 g of the substance to be examined to 10.0 mL with *water R*.

Test solution (b) Dilute 1.0 mL of test solution (a) to 20.0 mL with *water R*.

Reference solution (a) Dilute 1.0 mL of test solution (a) to 200.0 mL with *water R*.

Reference solution (b) Dissolve 10.0 mg of *raffinose pentahydrate R* (impurity A) and 14.0 mg of *glucose R* (impurity C) in *water R* and dilute to 50.0 mL with the same solvent. To 1.0 mL of the solution add 26.0 mg of *sucrose CRS* and dilute to 2.0 mL with *water R*.

Reference solution (c) Dissolve 33.0 mg of *sucrose CRS* in *water R* and dilute to 5.0 mL with the same solvent. Dilute 1.0 mL of the solution to 10.0 mL with *water R*.

Precolumn:

— *size*: *l* = 0.03 m, Ø = 8.0 mm;

— *stationary phase*: *strong cation-exchange resin (calcium form) R* (9 µm).

Column:

— *size*: *l* = 0.30 m, Ø = 7.8 mm;

— *stationary phase*: *strong cation-exchange resin (calcium form) R* (9 µm);

— *temperature*: 80 ± 1 °C.

Mobile phase *water for chromatography R*.

Flow rate 0.5 mL/min.

Detection Differential refractometer maintained at a constant temperature (about 40 °C).

Injection 10 µL of test solution (a) and reference solutions (a) and (b).

Run time 3 times the retention time of sucrose.

Identification of impurities Use the chromatogram obtained with reference solution (b) to identify the peaks due to impurities A (or B) and C.

Impurity A is present in sucrose obtained from sugar beet and impurity B is present in sucrose obtained from sugar cane.

Relative retention With reference to sucrose (retention time = about 10 min): impurities A and B = about 0.9; impurity C = about 1.2.

System suitability Reference solution (b):

— *peak-to-valley ratio*: minimum 2.5, where H_p = height above the baseline of the peak due to impurity A and H_v = height above the baseline of the lowest point of the curve separating this peak from the peak due to sucrose; minimum 2.5, where H_p = height above the baseline of the peak due to impurity C and H_v = height above the baseline of the lowest point of the curve separating this peak from the peak due to sucrose.

Calculation of percentage contents:

— for each impurity, use the concentration of sucrose in reference solution (a).

Limits:

— *impurity C*: maximum 0.7 per cent;

— *unspecified impurities*: for each impurity, maximum 0.5 per cent;

— *total*: maximum 2.0 per cent;

— *reporting threshold*: 0.05 per cent.

Sulfites

Maximum 10 ppm, calculated as SO_2.

Determine the sulfites content by a suitable enzymatic method based on the following reactions. Sulfite is oxidised by sulfite oxidase to sulfate and hydrogen peroxide which in turn is reduced by nicotinamide-adenine dinucleotide-peroxidase in the presence of reduced nicotinamide-adenine dinucleotide (NADH). The amount of NADH oxidised is proportional to the amount of sulfite.

Test solution Dilute 4.0 g of the substance to be examined to 10.0 mL with freshly prepared *distilled water R*.

Reference solution To 4.0 g of the substance to be examined add 0.5 mL of *sulfite standard solution (80 ppm SO_2) R* and dilute to 10.0 mL with freshly prepared *distilled water R*.

Blank solution Freshly prepared *distilled water R*.

Separately introduce 2.0 mL each of the test solution, the reference solution and the blank solution into 10 mm cuvettes and add the reagents as described in the instructions in the kit for sulfite determination. Measure the absorbance (*2.2.25*)

at the absorption maximum at about 340 nm before and at the end of the reaction time and subtract the value obtained with the blank.

The absorbance difference of the test solution is not greater than half the absorbance difference of the reference solution.

Dry matter

Determine the refractive index (2.2.6). Use Table 2797.-2 and interpolate the values if necessary.

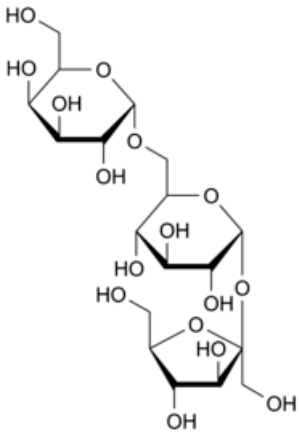
Table 2797.-2

Dry matter (per cent *m/m*)	
65	1.453478
66	1.455839
67	1.458217
68	1.460613
69	1.463026

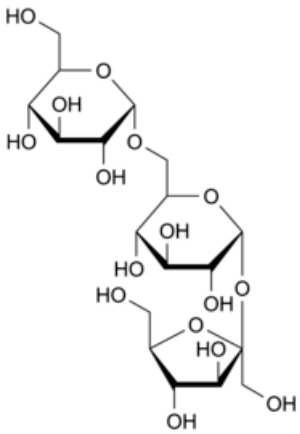
IMPURITIES

Specified impurities C.

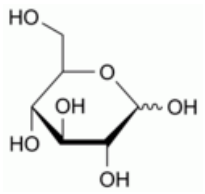
Other detectable impurities (the following substances would, if present at a sufficient level, be detected by one or other of the tests in the monograph. They are limited by the general acceptance criterion for other/unspecified impurities. It is therefore not necessary to identify these impurities for demonstration of compliance. See also 5.10. Control of impurities in substances for pharmaceutical use) A, B, D.



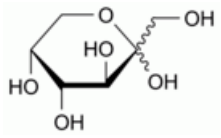
A. β-D-fructofuranosyl α-D-galactopyranosyl-(1→6)-α-D-glucopyranoside (raffinose),



B. β-D-fructofuranosyl α-D-glucopyranosyl-(1→6)-α-D-glucopyranoside (theanderose),



C. D-glucopyranose (glucose),



D. D-*arabino*-hex-2-ulopyranose (fructose).

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