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Polydextrose

CAS RN®: 68424-04-4.

DEFINITION

Polydextrose is a randomly branched polymer prepared by melting and subsequent condensation of the ingredients, which consist of approximately 90 parts dextrose, 10 parts sorbitol, and up to 1 part citric acid or 0.1 part phosphoric acid. The 1,6-glycosidic linkage predominates in the polymer but other linkages are present. It contains NLT 90.0% of dextrose polymer units, calculated on the anhydrous and ash-free basis. It contains small quantities of free dextrose, sorbitol, and 1,6-anhydro-D-glucose (levoglucosan), with traces of citric acid or phosphoric acid.

IDENTIFICATION

• **A.** To 1 drop of a solution (1 in 10), add 4 drops of 5% phenol solution, then rapidly add 15 drops of sulfuric acid TS: a deep yellow to orange color is produced.

• **B.** With vigorous swirling, add 1 mL of acetone to 1 mL of a solution (1 in 10): the solution remains clear.

• **C.** With vigorous swirling, add 2 mL of acetone to the solution obtained in *Identification B*: a heavy, milky turbidity develops immediately.

• **D.** To 1 mL of a solution (1 in 50), add 4 mL of alkaline cupric citrate TS. Boil vigorously for 2–4 min. Remove from heat, and allow the precipitate (if any) to settle: the supernatant is blue or blue-green.

Add the following:

▲• **E. Chromatographic Identity**

Analysis: Proceed as directed in the *Assay*.

Acceptance criteria: The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.▲ (1S (NF36))

ASSAY

Change to read:

• **Procedure**

Mobile phase: 0.001 N sulfuric acid. Pass this solution through a filter of 0.5-µm pore size, and degas.

Standard solution: 4.0 mg/mL of USP Polydextrose RS▲ (1S (NF36)) in *Mobile phase*

Sample solution: 4.0 mg/mL of ▲Polydextrose in *Mobile phase*▲ (ERR 1-Jul-2018)

Chromatographic system

(See *Chromatography ⟨621⟩, System Suitability*.)

Mode: LC

Detector: Refractive index

Detector temperature: 35 ± 0.1°

Columns

Guard: 4.6-mm × 3.0-cm; packing L17

Analytical: 7.8-mm × 30-cm; packing L17

Flow rate: 0.6 mL/min

Injection volume: 20 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Relative standard deviation: NMT 2.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of dextrose polymer units in the portion of Polydextrose taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak response of dextrose polymer units from the *Sample solution*

r_S = peak response of dextrose polymer units from the *Standard solution*

C_S = concentration of USP Polydextrose RS in the *Standard solution* (mg/mL)

C_U = concentration of Polydextrose in the *Sample solution* (mg/mL)

Acceptance criteria: ▲NLT 90.0% on the anhydrous and ash-free basis▲ (ERR 1-Jul-2018)

IMPURITIES

• **Residue on Ignition ⟨281⟩:** NMT 0.3%

• **Limit of Lead**

[Note—Use reagent-grade chemicals with as low a lead content as is practicable, as well as high-purity water and gases. Before use in this analysis, rinse all glassware and plasticware twice with 10% nitric acid and twice with 10% hydrochloric acid, and then rinse them thoroughly with Purified Water.]

Matrix modifier solution: Prepare a solution in water containing 100.0 mg of dibasic ammonium phosphate per 10 mL of solution.

Lead nitrate stock solution: Dissolve 159.8 mg of lead nitrate in 100 mL of water to which has been added 1 mL of nitric acid, then dilute with water to 1000 mL. Prepare and store this solution in glass containers free from soluble lead salts.

Standard lead solution: On the day of use, dilute 10.0 mL of *Lead nitrate stock solution* with water to 100.0 mL. Each milliliter of *Standard lead solution* contains the equivalent of 10 µg of lead.

Standard solution A: 0.02 µg/mL of lead, from *Standard lead solution* in water

Standard solution B: 0.05 µg/mL of lead, from *Standard lead solution* in water

Standard solution C: 0.1 µg/mL of lead, from *Standard lead solution* in water

Standard solution D: 0.2 µg/mL of lead, from *Standard lead solution* in water

Standard solution E: 0.5 µg/mL of lead, from *Standard lead solution* in water

Sample solution: Transfer 1.0 g of Polydextrose, weighed and calculated on the anhydrous and ash-free basis, into a 10-mL volumetric flask, and dissolve in and dilute with water to volume.

Spiked sample solution: Transfer 1.0 g of Polydextrose, weighed and calculated on the anhydrous and ash-free basis, into a 10-mL volumetric flask, and dissolve in water. Add 100 µL of the *Standard lead solution*, and dilute with water to volume. This solution contains 0.1 µg/mL of added lead.

Instrumental conditions

(See *Atomic Absorption Spectroscopy ⟨852⟩*.)

Mode: Graphite furnace atomic absorption spectrophotometer, equipped with a pyrolytic tube with a platform

Analytical wavelength: Lead emission line of 283.3 nm

Lamp: A lead hollow-cathode lamp, using a slit width of 0.7 mm (set low) and a deuterium arc lamp for background correction

Autosampler

Sample volume: 10 µL

Alternative volume: 10 µL of *Matrix modifier solution*

Furnace program: For the temperature program, see *Table 1*.

Table 1

Step	Dry	Char	Atomize	Clean	Recharge
Temperature (°)	130	800	2400	2600	20
Ramp time (s)	20	20	0	1	2

Step	Dry	Char	Atomize	Clean	Recharge
Hold time (s)	40	40	6	5	20
Argon flow rate (mL/min)	300	300	50	300	300

Analysis

Samples: 10 µL of the *Matrix modifier solution* was added into each 10-µL aliquot of the five *Standard solutions*, a mixture of 10 µL of the *Matrix modifier solution* and 10 µL of the *Sample solution*, and a mixture of 10 µL of the *Matrix modifier solution* and 10 µL of the *Spiked sample solution*

Concomitantly determine the absorbances of the *Samples* using the *Instrumental conditions* described above. Plot the absorbance of each *Standard solution*, compensated for background correction, versus its content of lead, in µg/mL, and draw the best straight line fitting the five points. From this plot, determine the concentrations, C_T and C_{ST}, in µg/mL, of lead in the *Sample solution* and the *Spiked sample solution*, respectively.

Calculate the percentage recovery taken:

$$\text{Result} = [(C_{ST} - C_T)/A] \times 100$$

A = quantity of lead added to the *Spiked sample solution*, 0.1 µg/mL

Calculate the content, in µg/g, of lead in the portion of Polydextrose taken:

$$\text{Result} = (C_T/W) \times V$$

W = weight of Polydextrose taken to prepare the *Sample solution* (g)

V = volume of the *Sample solution*, 10 mL

Acceptance criteria: NMT 0.5 µg/g. The recovery is 80%–120%.

- **Organic Impurities, Procedure 1: Limit of 5-Hydroxymethylfurfural and Related Compounds**

Sample solution: 1.0 g of Polydextrose, weighed and calculated on the anhydrous and ash-free basis, diluted with water to 100 mL

Analysis: Determine the absorbance of the *Sample solution* in a 1-cm quartz cell at 283 nm, with a suitable spectrophotometer, using water as the blank.

Calculate the percentage of 5-hydroxymethylfurfural and related compounds in the Polydextrose taken:

$$\text{Result} = 100 \times (V \times M_r \times A)/(M \times L \times W)$$

V = volume of the *Sample solution*, 0.1 L

M_r = molecular weight of 5-hydroxymethylfurfural, 126 g/mol

A = absorbance of the *Sample solution*

M = molar extinction coefficient of 5-hydroxymethylfurfural at a wavelength of 283 nm, 16,830 L/mol cm

L = path length of the spectrophotometer cell (cm)

W = weight of Polydextrose taken to prepare the *Sample solution* (g)

Acceptance criteria: NMT 0.1%

- **Organic Impurities, Procedure 2: Limit of Monomers**

Mobile phase, Sample solution, and **Chromatographic system:** Proceed as directed in the *Assay*.

Standard solution: 0.08 mg/mL each of USP 1,6-Anhydro-D-glucose RS and USP Sorbitol RS, and 0.16 mg/mL of USP Dextrose RS, in *Mobile phase*

System suitability

Sample: *Standard solution*

[Note—For relative retention times, see *Table 2*.]

Table 2

Name	Relative Retention Time
Dextrose (glucose)	0.7
Sorbitol	0.8
An isomer of 1,6-anhydro-D-glucose (D-anhydroglucose furanose form)	0.9
1,6-Anhydro-D-glucose (D-anhydroglucose pyranose form)	1.0

Suitability requirements

Resolution: NLT 1.0

Relative standard deviation: NMT 5.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Use peak response of USP 1,6-Anhydro-D-glucose RS in the *Standard solution* for calculation of percentage of the isomer of 1,6-anhydro-D-glucose in the *Sample solution*.

Calculate the percentage of each monomer in the portion of Polydextrose taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak response of the respective monomer from the *Sample solution*

r_S = peak response of the respective monomer from the *Standard solution*

C_S = concentration of the respective standard monomer in the *Standard solution* (mg/mL)

C_U = concentration of Polydextrose in the *Sample solution* (mg/mL)

Acceptance criteria: NMT 4.0% for 1,6-anhydro-D-glucose, NMT 4.0% for dextrose, and NMT 2.0% for sorbitol. [NOTE—In the case of 1,6-anhydro-D-glucose, the peak areas for the pyranose and furanose forms are combined.]

SPECIFIC TESTS

• **MOLECULAR WEIGHT LIMIT**

Mobile phase: Dissolve 35.0 g of sodium nitrate and 1.0 g of sodium azide in 100 mL of water. Dilute with water to 4 L. Pass through a filter of 0.45-µm pore size, and degas by applying an aspirator vacuum for 30 min. The resulting *Mobile phase* is 0.1 N sodium nitrate containing 0.025% sodium azide.

Standard solution: Transfer 20 mg each of USP Dextrose RS, stachyose, and 5800-, 23,700-, and 100,000-molecular weight (MW) pullulan standards into a 10-mL volumetric flask. Dissolve in and dilute with *Mobile phase* to volume. Pass through a syringe filter of 0.45-µm pore size into a suitable autosampler vial, and seal.

Sample solution: Transfer 50 mg of Polydextrose into a 10-mL volumetric flask. Dissolve in and dilute with *Mobile phase* to volume. Pass through a syringe filter of 0.45-µm pore size into a suitable autosampler vial, and seal.

Chromatographic system

(See *Chromatography ⟨621⟩, System Suitability*.)

Mode: LC

Detector: Refractive index set at a sensitivity of 4×10^{-6} refractive index units full scale and maintained at a temperature of 35 ± 0.1°

Column: 7.8-mm × 30-cm; packing L39

Column temperature: 45°

Flow rate: 0.8 mL/min

[NOTE—After installation of a new column, pump *Mobile phase* through the column overnight at a rate of 0.3 mL/min. Before calibration or analysis, increase the flow slowly over a 1-min period to 0.8 mL/min. Continue to pump *Mobile phase* through the column at this flow rate for at least 1 h before the first injection. Check the flow gravimetrically, and adjust it if necessary. Reduce the flow rate to about 0.1 mL/min when the system is not in use.]

Injection volume: 50 µL

System suitability

Sample: *Standard solution*

[NOTE—The retention times for each component determined on replicate injections agree within ±2 s.]

Chromatograph five replicate injections of the *Standard solution*, allowing 15 min between injections, and record the retention times of the components of the *Standard solution*.

Insert the average retention time along with the molecular weight of each component in the *Standard solution* into the calibration table of the molecular weight distribution software. Check the regression results for a cubic fit of the calibration points, and obtain a *Correlation coefficient R*, for the line.

Suitability requirements

Resolution: Dextrose and stachyose are baseline resolved from one another and from the 5800-MW pullulan standard.

[NOTE—Prominent negative baseline valleys are usually observed between the peaks for the 5800-; 23,700-; and 100,000-MW pullulan standards.]

Correlation coefficient R: NLT 0.9999

Analysis

Samples: *Standard solution* and *Sample solution*

Use the molecular weight distribution software of the data reduction system to generate a molecular weight distribution plot of Polydextrose.

Acceptance criteria: No measurable peak above a molecular weight of 22,000 is found.

• **pH** ⟨791⟩**:** 2.5–5.0, in a solution (1 in 10)

• **WATER DETERMINATION** ⟨921⟩, *Method I***:** NMT 4.0%. Use a mixture of Hydranal solvent and Hydranal formamide dry (2:1) as a solvent. Perform the titration at 50° in a jacketed beaker.

ADDITIONAL REQUIREMENTS

• **PACKAGING AND STORAGE:** Preserve in tight, light-resistant containers. Store in a cool and dry place.

• **USP REFERENCE STANDARDS** ⟨11⟩

USP 1,6-Anhydro-D-glucose RS
USP Dextrose RS
USP Polydextrose RS
USP Sorbitol RS

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
POLYDEXTROSE	Documentary Standards Support	CE2020 Complex Excipients
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	CE2020 Complex Excipients

Chromatographic Database Information: Chromatographic Database

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