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Hydrogenated Polydextrose

DEFINITION

Hydrogenated Polydextrose is obtained by transition metal catalytic hydrogenation of Polydextrose in aqueous solution. It contains NLT 90.0% of dextrose polymer units, calculated on the anhydrous and ash-free basis. The polymer chain end groups are mainly sorbitol-terminated.

IDENTIFICATION

- **A.**

  Sample: 1 drop of a solution (1 in 10)

  Analysis: Add 4 drops of 5% phenol solution to the *Sample* and then rapidly add 15 drops of sulfuric acid TS.

  Acceptance criteria: A deep yellow to orange color is produced.

- **B.**

  Sample: 1 mL of a solution (1 in 10)

  Analysis: With vigorous swirling, add 1 mL of acetone to the *Sample*.

  Acceptance criteria: The solution remains clear.

- **C.**

  Sample: Solution obtained in *Identification B*

  Analysis: With vigorous swirling, add 2 mL of acetone to the *Sample*.

  Acceptance criteria: A heavy, milky turbidity develops immediately.

- **D.**

  Sample: 1 mL of a solution (1 in 50)

  Analysis: Add 4 mL of alkaline cupric citrate TS to the *Sample*. Boil vigorously for 2–4 min. Remove from heat, and allow the precipitate (if any) to settle.

  Acceptance criteria: The supernatant is blue or blue-green.

- **E.** Meets the requirements for dextrose in *Organic Impurities, Procedure 2, Limit of Monomers*

Add the following:

▲- **F. 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*.▲ (NF 1-May-2023)

ASSAY

Change to read:

- **Procedure**

  Mobile phase: 0.001 N sulfuric acid, ▲prepared as follows. Cautiously transfer 3 mL of sulfuric acid into a 100-mL volumetric flask containing approximately 80 mL of water, dissolve, and dilute with water to volume. Pipet 1 mL of the resulting solution into a 1000-mL volumetric flask, and dilute with water to volume. Proportionally larger volumes of the second dilution may be prepared if desired.▲ (NF 1-May-2023)

  Standard solution: 4.0 mg/mL of ▲USP Hydrogenated Polydextrose RS▲ (NF 1-May-2023) in *Mobile phase*

  Sample solution: 4.0 mg/mL of Hydrogenated Polydextrose in *Mobile phase*

  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*. ▲[Note—Integrate and include smaller peaks eluted on the tailing of the Hydrogenated Polydextrose to be included as a part of the *Assay*.]▲ (NF 1-May-2023)

Calculate the percentage of dextrose polymer units in the portion of Hydrogenated 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 Hydrogenated Polydextrose RS▲ (NF 1-May-2023) in the *Standard solution* (mg/mL)

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

Acceptance criteria: NLT 90.0% on the anhydrous and ash-free basis

IMPURITIES

- **Residue on Ignition 〈281〉:** NMT 0.3%
- **Limit of Lead**

[Note—Use reagent-grade chemicals with a lead content of as low as possible, 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: 10.0 mg/mL of dibasic ammonium phosphate

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 the *Lead nitrate stock solution* with water to 100.0 mL. Each milliliter of the *Standard lead solution* contains the equivalent of 10 µg of lead.

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

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

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

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

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

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

Spiked sample solution: Transfer 1.0 g of Hydrogenated 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

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

Analytical wavelength: Lead emission line of 283.3 nm

Autosampler

Sample volume: 10 µL

Alternative volume: 10 µL of *Matrix modifier solution*

Furnace program: See the temperature program in *Table 1*.

Table 1

Step	Dry	Char	Atomize	Clean	Recharge
Temperature (°)	130	800	2400	2600	20
Ramp time (s)	20	20	0	1	2
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 of the 10-µL aliquots of the 5 *Standard solutions* (*Standard solutions A–E*), 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. 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 Hydrogenated Polydextrose taken:

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

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

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

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

Change to read:

- **LIMIT OF NICKEL**

▲**Digest solution:** Add 50 mL of nitric acid, ultratrace to a 1000-mL volumetric flask containing 500 mL of water. Mix and dilute with water to volume.

Nickel standard stock solution: 10 µg/mL of ICP Nickel (or multi-element) standard in 5% nitric acid solution. [NOTE—Single-element and multi-element ICP standard solutions are commercially available.[1]]

Standard solution A: Pipet 1 mL from the *Nickel standard stock solution* to a 100-mL volumetric flask. Dilute with *Digest solution* to volume and mix. This solution contains 0.1 µg/mL of nickel.

Standard solution B: Pipet 2 mL from the *Nickel standard stock solution* to a 100-mL volumetric flask. Dilute with *Digest solution* to volume and mix. This solution contains 0.2 µg/mL of nickel.

Standard solution C: Pipet 5 mL from the *Nickel standard stock solution* to a 100-mL volumetric flask. Dilute with *Digest solution* to volume and mix. This solution contains 0.5 µg/mL of nickel.

Sample solution: Transfer 5 g of Hydrogenated Polydextrose into a 100-mL volumetric flask, and dissolve in and dilute with *Digest solution* to volume.

Blank solution: Use a fresh aliquot of the *Digest solution*.

Chromatographic system

(See *Plasma Spectrochemistry ⟨730⟩*.)

Mode: ICP–OES

Emission wavelength: 352.4 nm for nickel. Set the sample read time and other instrument parameters as appropriate or as recommended by the instrument manufacturer.

System suitability

Samples: *Standard solution A*, *Standard solution B*, and *Blank solution*

Suitability requirements

[Note—Instrument performance must be verified to conform to the manufacturer's specifications for resolution and sensitivity. Before analyzing samples, the instrument must pass a suitable performance check.]

Correlation coefficient: NLT 0.995, determined from the calibration curve constructed in the *Analysis*

Analysis

Samples: *Standard solutions A–C* and *Blank solution*

[Note—The following analysis is described for one type of ICP–OES instrument. If a different ICP–OES instrument is used, follow the instrument manufacturer's recommendations for operation.]

Take 3 replicate scans with the integration set as recommended by the instrument manufacturer. Follow the instrument manufacturer's recommendations for delivering the sample. Flush the samples through the system before analysis. Program a read delay into the sampling routine to allow for fluid flow equilibration after the flush, before the first analytical read of the sample. Between samples, wash the pumping system by pushing the *Blank solution*.

Calibration curve: Generate the calibration curve using the *Blank solution* and *Standard solutions A–C* as follows. Construct a calibration curve by plotting the corrected nickel intensity versus the known concentrations, in µg/mL, of the nickel. Similarly, analyze the *Sample solution*. Plot the intensity of the emission of the *Sample solution* on the calibration curve. Determine the concentration of nickel (*C*), in µg/mL, in the *Sample solution* through the calibration curve.

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

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

V = volume of the *Sample solution*, 100 mL

C = concentration of nickel in the *Sample solution* (µg/mL)

W = weight of Hydrogenated Polydextrose (g) in the *Sample solution*▲ (NF 1-May-2023)

Acceptance criteria: NMT 2 µg/g

Organic Impurities

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

Sample solution: 1.0 g of Hydrogenated 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 portion of Hydrogenated Polydextrose taken:

$$\text{Result} = (V \times M_r \times A)/(\varepsilon_{283} \times L \times W) \times 100$$

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*

ε_{283} = molar extinction coefficient of 5-hydroxymethylfurfural at a wavelength of 283 nm, 16,830 $\text{L} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$

L = path length of the spectrophotometer cell (cm)

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

Acceptance criteria: NMT 0.1%

- **Procedure 2: Limit of Monomers**

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

Standard solution: 0.08 mg/mL of each for USP 1,6-Anhydro-D-glucose RS and USP Sorbitol RS and 0.04 mg/mL of USP Dextrose RS in *Mobile phase*

System suitability

Sample: *Standard solution*

[Note—See the relative retention times in *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 (levoglucosan) (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 the peak response of USP 1,6-Anhydro-D-glucose RS in the *Standard solution* for calculation of the percentage of the isomer of 1,6-anhydro-D-glucose in the *Sample solution*.

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

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

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

r_S = peak response for 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 Hydrogenated Polydextrose in the *Sample solution* (mg/mL)

Acceptance criteria

[Note—In the case of 1,6-anhydro-D-glucose, the peak areas for the pyranose and furanose forms are combined.]

1,6-Anhydro-D-glucose: NMT 4.0%

Sorbitol: NMT 5.75%

Dextrose: NMT 0.25%

SPECIFIC TESTS

Change to read:

- **Molecular Weight Limit**

▲[Note—Molecular weights (Mp) for the standards can be selected as available commercially while maintaining the required calibration range.]▲ (NF 1-May-2023)

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 or finer 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 or finer pore size into a suitable autosampler vial, and seal.

Sample solution: Transfer 50 mg of Hydrogenated 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 or finer pore size into a suitable autosampler vial, and seal.

Chromatographic system

(See Chromatography ⟨621⟩, System Suitability.)

Mode: LC

Detector: Refractive index ▲▲ (NF 1-May-2023) 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*

Chromatograph 5 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

Retention time: The retention times for each component determined on replicate injections agree within ±2 s.

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 Hydrogenated Polydextrose.

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

- **pH ⟨791⟩:** 5.0–7.0, in a solution (1 in 10)

Change to read:

- **Water Determination ⟨921⟩, *Method I*:** ▲[Note—Temperature can be adjusted depending on the titrator's capability.]▲ (NF 1-May-2023) 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.

Change to read:

- **USP Reference Standards ⟨11⟩**
  USP 1,6-Anhydro-d-glucose RS
  USP Dextrose RS
  ▲USP Hydrogenated Polydextrose RS▲ (NF 1-May-2023)
  USP Sorbitol RS

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[1] The multi-element standard can be purchased as BDH Aristar ICP standard cat. no. 89800-608, or equivalent.

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

Topic/Question	Contact	Expert Committee
HYDROGENATED 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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