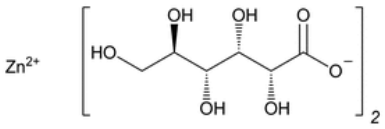
Printed on: Thu Nov 24 2022, 08:29:21 am
Printed by: XU SAI NAN
Status: Not Yet Official on 24-Nov-2022
Official Date: 01-Jun-2023
Document Type: USP & DS
DocId: GUID-1B0EC0AB-C4CA-4FF6-AB18-C8711BE65241_3_en-US
DOI: https://doi.org/10.31003/USPNF_M89660_03_01
DOI Ref: 2bq4p

Zinc Gluconate



$C_{12}H_{22}O_{14}Zn$ 455.67
Bis(D-gluconato-O^1,O^2) zinc;
Zinc D-gluconate (1:2) CAS RN®: 4468-02-4.

DEFINITION

Zinc Gluconate contains NLT 97.0% and NMT 102.0% of zinc gluconate ($C_{12}H_{22}O_{14}Zn$), calculated on the anhydrous basis.

IDENTIFICATION

- **A. Identification Tests—General, *Zinc* ⟨191⟩:** A 100-mg/mL solution meets the requirements.
- **B. Thin-Layer Chromatography**

Standard solution: 10 mg/mL of USP Potassium Gluconate RS

Sample solution: 10 mg/mL of Zinc Gluconate, heating in a water bath at 60°, if necessary, to dissolve

Chromatographic system

(See *Chromatography* ⟨621⟩, *Thin-Layer Chromatography*.)

Mode: TLC

Adsorbent: 0.25-mm layer of chromatographic silica gel

Application volume: 5 µL

Developing solvent system: Alcohol, ethyl acetate, ammonium hydroxide, and water (50:10:10:30)

Spray reagent: Dissolve 2.5 g of ammonium molybdate in 50 mL of 2 N sulfuric acid in a 100-mL volumetric flask, add 1.0 g of ceric sulfate, swirl to dissolve, and dilute with 2 N sulfuric acid to volume.

Analysis

Samples: *Standard solution* and *Sample solution*

Develop the chromatogram until the solvent front has moved about three-fourths of the length of the plate. Remove the plate from the chamber, and dry at 110° for 20 min. Allow to cool, and spray with the *Spray reagent*. Heat the plate at 110° for about 10 min.

Acceptance criteria: The principal spot of the *Sample solution* corresponds in color, size, and R_F value to that of the *Standard solution*.

ASSAY

- **Procedure**

Sample: 700 mg of Zinc Gluconate

Blank: 100 mL of water

Titrimetric system

(See *Titrimetry* ⟨541⟩.)

Mode: Direct titration

Titrant: 0.05 M edetate disodium VS

Endpoint detection: Visual

Analysis: Dissolve the *Sample* in 100 mL of water. Add 5 mL of ammonia–ammonium chloride buffer TS and 0.1 mL of eriochrome black TS. Titrate with *Titrant* until the solution is deep blue in color. Perform a blank determination.

Calculate the percentage of zinc gluconate ($C_{12}H_{22}O_{14}Zn$) in the *Sample* taken:

$$\text{Result} = \{[(V_S - V_B) \times M \times F]/W\} \times 100$$

V_S = *Titrant* volume consumed by the *Sample* (mL)

V_B = *Titrant* volume consumed by the *Blank* (mL)

M = *Titrant* molarity (mmol/mL)

F = equivalency factor, 455.7 mg/mmol

W = *Sample* weight (mg)

Acceptance criteria: 97.0%–102.0% on the anhydrous basis

IMPURITIES

• **Chloride and Sulfate,** *Chloride* ⟨221⟩

Standard solution: 0.7 mL of 0.020 N hydrochloric acid

Sample: 1.0 g

Acceptance criteria: NMT 0.05%

• **Chloride and Sulfate,** *Sulfate* ⟨221⟩

Standard solution: 1.0 mL of 0.020 N sulfuric acid

Sample: 2.0 g

Acceptance criteria: NMT 0.05%

Change to read:

• ▲**Arsenic** ⟨211⟩**,** *Procedures, Procedure 1*▲ (CN 1-Jun-2023)

Test preparation: 1 g in 35 mL of water

Acceptance criteria: NMT 3 ppm

• **Limit of Lead**

[Note—For the preparation of all aqueous solutions and for the rinsing of glassware before use, use water that has been passed through a strong-acid, strong-base, mixed-bed ion-exchange resin. Select all reagents to have as low a content of lead as practicable, and store all reagent solutions in containers of borosilicate glass. Cleanse glassware before use by soaking in warm 8 N nitric acid for 30 min and by rinsing with deionized water.]

Ascorbic acid–sodium iodide solution: 100 mg/mL of ascorbic acid and 192.5 mg/mL of sodium iodide

Trioctylphosphine oxide solution: 50 mg/mL of trioctylphosphine oxide in 4-methyl-2-pentanone.

[**Caution—**This solution causes irritation. Avoid contact with eyes, skin, and clothing. Take special precautions in disposing of unused portions of solutions to which this reagent is added.]

Standard solution: Transfer 5.0 mL of lead nitrate stock solution TS, to a 100-mL volumetric flask, and dilute with water to volume. Transfer 2.0 mL of the resulting solution to a 50-mL volumetric flask, and add 10 mL of 9 N hydrochloric acid and 10 mL of water. Add 20 mL of *Ascorbic acid–sodium iodide solution* and 5.0 mL of *Trioctylphosphine oxide solution*. Shake for 30 s, and allow to separate. Add water to bring the organic solvent layer into the neck of the flask, shake again, and allow to separate. The organic layer is the *Standard solution*, and it contains 2 µg/mL of lead.

Sample solution: To a 50-mL volumetric flask add 1.0 g of Zinc Gluconate , 10 mL of 9 N hydrochloric acid, 10 mL of water, 20 mL of *Ascorbic acid–sodium iodide solution*, and 5.0 mL of *Trioctylphosphine oxide solution*. Shake for 30 s, and allow to separate. Add water to bring the organic solvent layer into the neck of the flask, shake again, and allow to separate. The organic layer is the *Sample solution*.

Blank: To a 50-mL volumetric flask add 10 mL of 9 N hydrochloric acid, 10 mL of water, 20 mL of *Ascorbic acid–sodium iodide solution*, and 5.0 mL of *Trioctylphosphine oxide solution*. Shake for 30 s, and allow to separate. Add water to bring the organic solvent layer into the neck of the flask, shake again, and allow to separate. The organic layer is the *Blank*, and it contains 0 µg/mL of lead.

Instrumental conditions

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

Mode: Atomic absorption spectrophotometry

Analytical wavelength: 283.3 nm

Lamp: Lead hollow-cathode

Flame: Air–acetylene

System suitability

Samples: *Standard solution* and *Blank*

Suitability requirements: The absorbance of the *Standard solution* and the absorbance of the *Blank* are significantly different.

Analysis

Samples: *Standard solution*, *Sample solution*, and *Blank*

Concomitantly determine the absorbances of the *Blank*, *Standard solution*, and the *Sample solution*. Use the *Blank* to set the instrument to zero.

Acceptance criteria: NMT 10 ppm: the absorbance of the *Sample solution* does not exceed that of the *Standard solution*.

• **Limit of Cadmium**

Standard stock solution: 0.1372 mg/mL of cadmium nitrate

Standard solution: Pipet 25 mL of the *Standard stock solution* into a 100-mL volumetric flask, add 1 mL of hydrochloric acid, dilute with water to volume, and mix. It contains 12.5 µg/mL of cadmium (Cd).

Sample stock solution: Transfer 10.0 g of Zinc Gluconate into a 50-mL volumetric flask, and dissolve in and dilute with water to volume.

Sample solution A: Transfer 5.0 mL of the *Sample stock solution* to a 25-mL volumetric flask, and dilute with water to volume. This solution contains 0 µg/mL of added cadmium from the *Standard solution*.

Sample solution B: Transfer 5.0 mL of the *Sample stock solution* to a 25-mL volumetric flask, add 2.0 mL of the *Standard solution*, and dilute with water to volume. This solution contains 1.0 µg/mL of added cadmium from the *Standard solution*.

Sample solution C: Transfer 5.0 mL of the *Sample stock solution* to a 25-mL volumetric flask, add 4.0 mL of the *Standard solution*, and dilute with water to volume. This solution contains 2.0 µg/mL of added cadmium from the *Standard solution*.

Blank: Water

Instrumental conditions

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

Mode: Atomic absorption spectrophotometry

Analytical wavelength: 228.8 nm

Lamp: Cadmium hollow-cathode

Flame: Air–acetylene

Analysis

Samples: *Sample solution A*, *Sample solution B*, and *Sample solution C*

Determine the absorbances. Correct the absorbance values of *Sample solution A*, *Sample solution B*, and *Sample solution C* from that of the *Blank*. Plot the corrected absorbances of *Sample solution A*, *Sample solution B*, and *Sample solution C* versus their added cadmium concentrations, in µg/mL. Draw the straight line best fitting the three points, and extrapolate the line until it intercepts the concentration axis. From the intercept, determine the amount, in µg/mL, of cadmium in *Sample solution A*.

Calculate the content of cadmium in the portion of Zinc Gluconate taken:

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

C = concentration of cadmium in *Sample solution A* (µg/mL), determined from the intercept of the linear regression line

V = volume of solvent taken to prepare *Sample solution A* (mL)

W = weight of Zinc Gluconate taken to prepare *Sample solution A* (g)

Acceptance criteria: NMT 5 ppm

- **Reducing Substances**

Sample: 1.0 g of Zinc Gluconate

Blank: 10 mL of water

Titrimetric system

(See *Titrimetry ⟨541⟩*.)

Mode: Residual titration

Titrant 0.1 N iodine VS

Back titrant: 0.1 N sodium thiosulfate VS

Endpoint detection: Visual

Analysis: Transfer the *Sample* to a 250-mL conical flask, dissolve in 10 mL of water, and add 25 mL of alkaline cupric citrate TS. Cover the flask, boil gently for 5 min, accurately timed, and cool rapidly to room temperature. Add 25 mL of 0.6 N acetic acid, 10.0 mL of *Titrant*, and 10 mL of 3 N hydrochloric acid, and titrate with *Back titrant*, adding 3 mL of starch TS as the endpoint is approached. Perform the blank determination. Calculate the percentage of reducing substances (as dextrose) in the *Sample* taken:

$$\text{Result} = \{[(V_B - V_S) \times N \times F]/W\} \times 100$$

V_B = *Back titrant* volume consumed by the *Blank* (mL)

V_S = *Back titrant* volume consumed by the *Sample* (mL)

N = *Back titrant* normality (mEq/mL)

F = equivalency factor, 27 mg/mEq

W = *Sample* weight (mg)

Acceptance criteria: NMT 1.0%

SPECIFIC TESTS

- **pH** ⟨791⟩

  Sample solution: 10 mg/mL

  Acceptance criteria: 5.5–7.5

- **Water Determination,** *Method Ib* ⟨921⟩**:** NMT 11.6%

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in well-closed containers.
- **USP Reference Standards** ⟨11⟩

  USP Potassium Gluconate RS

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

Topic/Question	Contact	Expert Committee
ZINC GLUCONATE	Sandeep Putty Senior Scientist I	NBDS2020 Non-botanical Dietary Supplements

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Chromatographic Database Information: Chromatographic Database

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Current DocID: GUID-1B0EC0AB-C4CA-4FF6-AB18-C8711BE65241_3_en-US

DOI: https://doi.org/10.31003/USPNF_M89660_03_01

DOI ref: 2bq4p