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Stannous Fluoride Gel

DEFINITION

Stannous Fluoride Gel contains NLT 95.0% and NMT 115.0% of the labeled amount of stannous fluoride (SnF_2) in a suitable medium containing a suitable viscosity-inducing agent.

[Note—If Glycerin is used as the medium in the preparation of this Gel, use Glycerin that has a low water content; that is, Glycerin having a specific gravity of NLT 1.2607, corresponding to a concentration of 99.5%.]

IDENTIFICATION

Change to read:

• **A.** ▲The retention time of the fluoride peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.▲

(USP 1-Aug-2022)

• **B.**

Sample solution: Nominally 1 mg/mL of stannous fluoride from Gel in water

Analysis: On a spot plate, mix 2 drops of *Sample solution* with 2 drops of silver nitrate TS.

Acceptance criteria: A brown-black precipitate is formed.

• **C.**

Sample solution: Nominally 1 mg/mL of stannous fluoride from Gel in water

Analysis: Add 1 drop of *Sample solution* to 2 drops of mercuric chloride TS.

Acceptance criteria: A white, silky precipitate is formed. On further addition of the *Sample solution*, a brown-black precipitate is formed.

ASSAY

Change to read:

• **Procedure**

▲[Note—Store all solutions in plastic containers. It is recommended to use plastic HPLC vials. Use water with a resistivity of NLT 18 megohm-cm to prepare the solutions.]

Mobile phase: 15 mM potassium hydroxide in water. [Note—*Mobile phase* can be generated electrolytically using an automatic eluant generator.]

System suitability solution: 2.0 µg/mL of USP Sodium Fluoride RS and 1.0 µg/mL of USP Sodium Acetate RS in water

Standard solution: 2.0 µg/mL of USP Sodium Fluoride RS in water

Sample solution: Nominally 3.7 µg/mL of stannous fluoride in water prepared as follows. Transfer an accurately weighed portion of Gel to a suitable volumetric flask, add about 60% of the flask volume of water, and dissolve with shaking. Dilute with water to volume.

Chromatographic system

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

Mode: LC

Detector: Conductivity with suppression

Columns

Guard: 4.0-mm × 5-cm; 13-µm packing L120. [Note—Alternatively, a 4.0-mm × 0.5-cm column that contains 4.6-µm packing L91 may be used.]

Analytical: 4.0-mm × 25-cm; 7.5-µm packing L113. [Note—Alternatively, a 4.0-mm × 25-cm column that contains 4.6-µm packing L91 may be used.]

Column temperature: 40°

Flow rate: 1.0 mL/min

Injection volume: 20 µL

Run time: NLT 6 times the retention time of fluoride

System suitability

Samples: *System suitability solution* and *Standard solution*

[NOTE—The relative retention times for the fluoride and acetate ions are 1.0 and 1.1, respectively.]

Suitability requirements

Resolution: NLT 1.5 between the fluoride and acetate ions, *System suitability solution*

Tailing factor: NMT 2.0 for the fluoride ion, *Standard solution*

Relative standard deviation: NMT 1.0% for the fluoride ion, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of stannous fluoride (SnF_2), P_{SnF_2}, in the portion of Gel taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times (M_{r1}/M_{r2}) \times 100$$

r_U = peak response of the fluoride ion from the *Sample solution*

r_S = peak response of the fluoride ion from the *Standard solution*

C_S = concentration of USP Sodium Fluoride RS in the *Standard solution* (µg/mL)

C_U = nominal concentration of stannous fluoride in the *Sample solution* (µg/mL)

M_{r1} = molecular weight of stannous fluoride, 156.71

M_{r2} = molecular weight of sodium fluoride, 41.99▲ (USP 1-Aug-2022)

Acceptance criteria: 95.0%–115.0%

SPECIFIC TESTS

Change to read:

• **CONTENT OF TOTAL TIN**

Potassium chloride solution: 19.1 mg/mL of potassium chloride in water

Standard stock solution: ▲1.000▲ (USP 1-Aug-2022) mg/mL of tin prepared as follows. Transfer an appropriate amount of tin (Sn) to a suitable volumetric flask. Add hydrochloric acid to about 20% of the final volume of the flask, and swirl to dissolve. Add 20% of the flask volume of water into the flask, and allow to cool. Dilute with water to volume. ▲▲ (USP 1-Aug-2022)

Standard solution A: 50.0 µg/mL of tin and 191 µg/mL of potassium chloride in water from *Standard stock solution* and *Potassium chloride solution*

Standard solution B: 100.0 µg/mL of tin and 191 µg/mL of potassium chloride in water from *Standard stock solution* and *Potassium chloride solution*

Standard solution C: 150.0 µg/mL of tin and 191 µg/mL of potassium chloride in water from *Standard stock solution* and *Potassium chloride solution*

Sample solution: Nominally 0.132 mg/mL of stannous fluoride from a portion of Gel prepared as follows. Transfer an accurately weighed quantity of Gel, equivalent to about 132 mg of stannous fluoride, to a plastic beaker. Add 80 mL of water and 20 mL of hydrochloric acid, and mix. Transfer this mixture to a 1000-mL volumetric flask, add 10.0 mL of *Potassium chloride solution*, and dilute with water to volume. ▲▲ (USP 1-Aug-2022)

Blank: *Potassium chloride solution*, hydrochloric acid, and water (1:2:97)

Instrumental conditions

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

Mode: Atomic absorption spectrophotometer

Analytical wavelength: 235.5 nm

Lamp: Tin hollow-cathode

Flame: Nitrous oxide-acetylene oxidizing flame

Analysis

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

Use water to adjust the instrument to zero. Concomitantly determine the absorbance of *Standard solution A*, *Standard solution B*, *Standard solution C*, *Sample solution*, and *Blank*. Aspirate water before and after each determination. Correct the absorbances of *Standard solution A*, *Standard solution B*, *Standard solution C*, and the *Sample solution* by subtracting the absorbance of the *Blank*. Plot the corrected absorbances of *Standard solution A*, *Standard solution B*, and *Standard solution C* versus concentration, in µg/mL, of tin, and

draw the straight line best fitting the three plotted points. From the graph so obtained, determine the concentration (C), in µg/mL, of tin in the *Sample solution*.

Calculate the concentration of tin ▲(C_{Tin}), in µg/mg, in the labeled amount of stannous fluoride:▲ (USP 1-Aug-2022)

$$\text{Result} = C/C_U$$

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

C_U = nominal concentration of stannous fluoride in the *Sample solution* (mg/mL)

[NOTE—Use this value to calculate the percentage of stannous ▲tin▲ (USP 1-Aug-2022) for the test for the *Content of Stannous ▲Tin*.▲ (USP 1-Aug-2022)]

Change to read:

• **CONTENT OF STANNOUS ▲TIN▲** (USP 1-AUG-2022)

Solution A: 5 mg/mL of sodium hydroxide and 50 mg/mL of potassium iodide in oxygen-free water

Potassium iodide–iodate titrant: 0.1 N potassium iodide-iodate solution prepared as follows. In a 1000-mL volumetric flask, dissolve 3.567 g of potassium iodate, previously dried at 110° to constant weight, in 200 mL of *Solution A*. Dilute with oxygen-free water to volume, and mix. Standardize this solution by titrating a solution prepared from an accurately weighed quantity of reagent tin (Sn) and hydrochloric acid. Each milliliter of 0.1 N *Potassium iodide-iodate titrant* is equivalent to 5.935 mg of tin (Sn).

Sample: A suitable amount of Gel, ▲nominally▲ (USP 1-Aug-2022) equivalent to 80 mg of stannous fluoride

Titrimetric system

▲(See *Titrimetry* ⟨541⟩.)▲ (USP 1-Aug-2022)

Mode: Direct titration

Titrant: *Potassium iodide–iodate titrant*

Endpoint detection: Visual

Analysis: Transfer the *Sample* to a capped plastic vessel equipped for titration in an inert atmosphere. Add a plastic coated stirring bar, 20 mL of recently boiled 3 N hydrochloric acid, and 5 mL of potassium iodide TS. Close the vessel, purge the system with an oxygen-free inert gas, and titrate immediately with *Potassium iodide–iodate titrant*, adding 2 mL of starch TS as the endpoint is approached.

▲Calculate the percentage of stannous tin (Sn^{2+}) relative to the amount of stannous fluoride in the portion of Gel taken:

$$\text{Result} = [(V_S \times N_A \times F_1)/(P_{SnF_2} \times F_2 \times W)] \times 100$$

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

N_A = actual normality of the *Titrant* (mEq/mL)

F_1 = equivalency factor of stannous tin, 59.35 mg/mEq

P_{SnF_2} = percentage of the labeled amount of stannous fluoride, as determined in the *Assay* (%)

F_2 = conversion factor from percent to decimal, 0.01

W = nominal weight of stannous fluoride in the *Sample* taken (mg)

Calculate the percentage of stannous tin (Sn^{2+}) relative to the amount of total tin in the portion of Gel taken:

$$\text{Result} = [(V_S \times N_A \times F_1 \times F_2)/(C_{Tin} \times W)] \times 100$$

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

N_A = actual normality of the *Titrant* (mEq/mL)

F_1 = equivalency factor of stannous tin, 59.35 mg/mEq

F_2 = conversion factor, 1000 µg/mg

C_{Tin} = concentration of total tin, as determined in the *Content of Total Tin* test (µg/mg)

W = nominal weight of stannous fluoride in the *Sample* taken (mg)▲ (USP 1-Aug-2022)

Acceptance criteria: ▲NLT 68.2% of stannous fluoride and NLT 90.0% of total tin▲ (USP 1-Aug-2022)

• **Viscosity—Rotational Methods** ⟨912⟩

Sample: A portion of Gel

Analysis: Transfer the *Sample* to a suitable plastic container, insert the stopper securely, and allow to stand until the *Sample* is free from air bubbles. Place it in a water bath maintained at a temperature of 25 ± 0.5° until it adjusts to the temperature of the water bath (4 h or longer). Do not stir the *Sample* while it is in the bath. Remove the *Sample* from the bath, stir the *Sample* gently for 2 min, and without delay, using a rotational viscometer, determine the viscosity by using a spindle having a cylinder 1.27 cm in diameter and 0.16 cm high that is attached to a shaft 0.32 cm in diameter, with the distance from the top of the cylinder to the lower tip of the shaft being 2.54 cm and the immersion depth being 5.00 cm (No. 3 spindle). Operate the viscometer at 12 rpm, and record the scale reading at 1-min intervals for 4 min. Calculate the viscosity, in centipoises, by multiplying the scale reading by 100.

Acceptance criteria: 600–170,000 centipoises

• **pH** ⟨791⟩

Sample solution: A freshly prepared mixture with water (50:50)

Acceptance criteria: 2.8–4.0

ADDITIONAL REQUIREMENTS

Change to read:

• **Packaging and Storage:** Preserve in ▲tight▲ (USP 1-Aug-2022) containers. ▲Store at 15°–30°.▲ (USP 1-Aug-2022)

Change to read:

• **USP Reference Standards** ⟨11⟩

▲USP Sodium Acetate RS▲ (USP 1-Aug-2022)

USP Sodium Fluoride RS

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

Topic/Question	Contact	Expert Committee
STANNOUS FLUORIDE GEL	Documentary Standards Support	SM32020 Small Molecules 3
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM32020 Small Molecules 3

Chromatographic Database Information: Chromatographic Database

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