Status: Currently Official on 16-Feb-2025
Official Date: Official as of 01-Aug-2022
Document Type: USP Monographs
DocId: GUID-A47DBB12-4E42-4026-A00A-FF0E93320A2C_2_en-US
DOI: https://doi.org/10.31003/USPNF_M78000_02_01
DOI Ref: 38vj5

Stannous Fluoride

Change to read:

SnF_2 156.71

▲Tin (II) fluoride;

Difluoro-λ^2-stannane▲ (USP 1-Aug-2022) CAS RN®: 7783-47-3; UNII: 3FTR44B32Q.

Change to read:

DEFINITION

Stannous Fluoride contains NLT 71.2% of stannous tin ($Sn^{▲2+▲}$ (USP 1-Aug-2022)), and NLT 22.3% and NMT 25.5% of fluoride ($F^{▲-▲}$ (USP 1-Aug-2022)), calculated on the dried basis.

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, Procedure 2: Fluoride*.▲ (USP 1-Aug-2022)

• **B.**

Sample solution: 10 mg/mL of Stannous Fluoride in water

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

Acceptance criteria: A brown-black precipitate is formed.

• **C.**

Sample solution: 10 mg/mL of Stannous Fluoride in water

Analysis: Add 1 drop of the *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 1: 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 solution: 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*, and dilute with oxygen-free water to volume. Standardize this solution by titrating a solution prepared from an accurately weighed quantity of tin (Sn) and hydrochloric acid. Each milliliter of 0.1 N potassium iodide–iodate solution is equivalent to 5.935 mg of tin (Sn).

Sample: 250 mg of Stannous Fluoride

Titrimetric system

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

Mode: Direct titration

Titrant: *Potassium iodide–iodate solution*

Endpoint detection: Visual

Analysis: Transfer the *Sample* to a 500-mL conical flask, and add 300 mL of hot, recently boiled 3 N hydrochloric acid. While passing a stream of an oxygen-free inert gas over the surface of the liquid, swirl the flask to dissolve the *Sample*, and cool to room temperature. Add 5 mL of potassium iodide TS, and titrate in an inert atmosphere with *Titrant*, adding 3 mL of starch TS as the endpoint is approached.

▲Calculate the percentage of stannous tin (Sn^{2+}) in the portion of Stannous Fluoride taken:

$$\text{Result} = V_S \times N \times F \times (1/W) \times 100$$

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

N = actual *Titrant* concentration, in normality (mEq/mL)

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

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

Acceptance criteria: NLT 71.2% on the dried basis

Change to read:

• **Procedure 2: Fluoride**

▲[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: 3.7 µg/mL of Stannous Fluoride in water

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 fluoride (F^-) in the portion of Stannous Fluoride taken:

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

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 = concentration of Stannous Fluoride in the *Sample solution* (µg/mL)

A_r = atomic weight of fluoride, 19.00

M_r = molecular weight of sodium fluoride, 41.99▲ (USP 1-Aug-2022)

Acceptance criteria: 22.3%–25.5% on the dried basis

IMPURITIES

Change to read:

• **Antimony**

Solution A: 0.1 mg/mL of rhodamine B in 0.5 N hydrochloric acid

Standard stock solution: 0.275 mg/mL of antimony potassium tartrate in water

Standard solution: 2.75 µg/mL of antimony potassium tartrate in 6 N hydrochloric acid from the *Standard stock solution*

Sample solution: 20 mg/mL of Stannous Fluoride in 6 N hydrochloric acid

Instrumental conditions

▲(See *Ultraviolet-Visible Spectroscopy* ⟨857⟩.)▲ (USP 1-Aug-2022)

Mode: Vis

Analytical wavelength: 550 nm

Blank: Water

Analysis

Samples: *Standard solution* and *Sample solution*

Transfer 5 mL each of the *Standard solution* and the *Sample solution* into separate 125-mL separators, add 15 mL of hydrochloric acid and 1 g of ceric sulfate, and allow to stand for 5 min, with occasional shaking. Add 500 mg of hydroxylamine hydrochloride, and shake for 1 min. Add 15 mL of isopropyl ether into the mixture, shake for 30 s, and add 7 mL of water. Cool in a water bath at room temperature for 10 min, shake for 30 s, allow the layers to separate, and discard the aqueous phase. Add 20 mL of *Solution A*, shake for 30 s, and discard the aqueous layer. Decant the ether layer from the top of the separator, and centrifuge, if necessary, to obtain a clear solution. Concomitantly determine the absorbances of the ether solutions from the *Standard solution* and the *Sample solution*.

Acceptance criteria: The absorbance of the *Sample solution* is NMT that of the *Standard solution* (0.005%).

• **Water-Insoluble Substances**

Sample: 10 g of Stannous Fluoride

Analysis: Transfer the *Sample* to a 400-mL plastic beaker, add 200 mL of water, and stir with a plastic rod for 3 min, or until no more solid dissolves. Filter through a tared filtering crucible, and wash thoroughly, first with 10 mg/mL of ammonium fluoride solution, then with water. [Note—Prepare and use the filtering crucible in a well-ventilated hood.] Dry the residue at 105° for 4 h, cool, and weigh.

Acceptance criteria: NMT 0.2%

SPECIFIC TESTS

• **pH** ⟨791⟩

Sample solution: 4 mg/mL of Stannous Fluoride in water, freshly prepared

Acceptance criteria: 2.8–3.5

• **Loss on Drying** ⟨731⟩

Analysis: Dry at 105° for 4 h.

Acceptance criteria: NMT 0.5%

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in well-closed containers.

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	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

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. 47(2)

Current DocID: GUID-A47DBB12-4E42-4026-A00A-FF0E93320A2C_2_en-US

DOI: https://doi.org/10.31003/USPNF_M78000_02_01
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