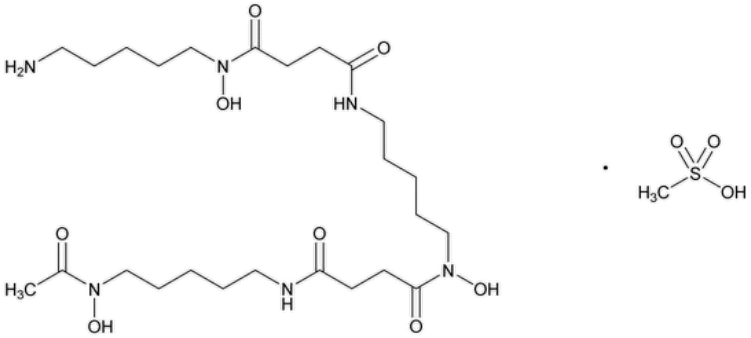
Status: Currently Official on 14-Feb-2025
Official Date: Official as of 01-May-2020
Document Type: USP Monographs
DocId: GUID-2DA07CF4-1552-473A-BC94-B87C1B7C97B5_4_en-US
DOI: https://doi.org/10.31003/USPNF_M22400_04_01
DOI Ref: ub8rr

Deferoxamine Mesylate



$C_{25}H_{48}N_6O_8 \cdot CH_4O_3S$ 656.79

Butanediamide, *N*′-[5-[[4-[[5-(acetylhydroxyamino)pentyl] amino]-1,4-dioxobutyl]hydroxyamino]pentyl]-*N*-(5-aminopentyl)-*N*-hydroxy-, monomethanesulfonate;
N-[5-[3-[(5-Aminopentyl)hydroxycarbamoyl]propionamido] pentyl]-3-[[5-(*N*-hydroxyacetamido)pentyl]carbamoyl]propionohydroxamic acid monomethanesulfonate (salt) CAS RN®: 138-14-7; UNII: V9TKO7EO6K.

DEFINITION

Deferoxamine Mesylate contains NLT 93.0% and NMT 102.0% of deferoxamine mesylate ($C_{25}H_{48}N_6O_8 \cdot CH_4O_3S$), calculated on the anhydrous basis.

IDENTIFICATION

Change to read:

• **A.** ▲Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*: **197K**▲ (CN 1-May-2020)

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

ASSAY

• **Procedure**

Solution A: 1.32 g/L of dibasic ammonium phosphate in water. Adjust with phosphoric acid to a pH of 3.0.

Solution B: Acetonitrile and *Solution A* (1:1)

Mobile phase: See *Table 1*.

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	88	12
20	80	20
35	57.5	42.5
35.1	88	12
40	88	12

Diluent: Acetonitrile and water (6:94)

Standard solution: 1.0 mg/mL of USP Deferoxamine Mesylate RS in *Diluent*

Sample solution: 1.0 mg/mL of Deferoxamine Mesylate in *Diluent*

Chromatographic system

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

Mode: LC

Detector: UV 220 nm

Column: 4.6-mm × 7.5-cm; 3.5-µm packing L1

Temperatures

Column: 32°

Autosampler: 5°

Flow rate: 1.5 mL/min

Injection volume: 20 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Relative standard deviation: NMT 0.73%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of deferoxamine mesylate ($C_{25}H_{48}N_6O_8 \cdot CH_4O_3S$) in the portion of Deferoxamine Mesylate taken:

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

r_U = peak response of deferoxamine from the *Sample solution*

r_S = peak response of deferoxamine from the *Standard solution*

C_S = concentration of the *Standard solution* (mg/mL)

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

Acceptance criteria: 93.0%–102.0% on the anhydrous basis

IMPURITIES

• **Residue on Ignition ⟨281⟩:** NMT 0.1%, 2.0 g being used

• **Chloride and Sulfate, *Chloride*⟨221⟩:** NMT 0.012%; a 1.2-g portion shows no more chloride than corresponds to 0.20 mL of 0.020 N hydrochloric acid.

• **Chloride and Sulfate, *Sulfate*⟨221⟩:** NMT 0.04%; a 0.5-g portion shows no more sulfate than corresponds to 0.20 mL of 0.020 N sulfuric acid.

• **Organic Impurities**

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

Standard stock solution: Use the *Standard solution*, prepared as directed in the *Assay*. [Note—USP Deferoxamine Mesylate RS contains impurity A as a minor component.]

Standard solution: 0.01 mg/mL of USP Deferoxamine Mesylate RS in *Diluent* from the *Standard stock solution*

System suitability

Samples: *Standard stock solution* and *Standard solution*

Suitability requirements

Resolution: NLT 2.0 between the impurity A and deferoxamine peaks, *Standard stock solution*

Relative standard deviation: NMT 5.0% for the deferoxamine peak, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of each impurity in the portion of Deferoxamine Mesylate taken:

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

r_U = peak response of each impurity from the *Sample solution*

r_S = peak response of deferoxamine from the *Standard solution*

C_S = concentration of the *Standard solution* (mg/mL)

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

Acceptance criteria: See *Table 2*.

[NOTE—The reporting level for impurities is 0.04%.]

Table 2

Name	Relative Retention Time	Acceptance Criteria, NMT (%)
Impurity A[a,b]	0.85–0.87	3.0[c]
Deferoxamine	1.0	—
Any unspecified impurity	—	1.0
Total impurities eluting before deferoxamine	—	5.0
Total impurities eluting after deferoxamine	—	2.0

[a] Des-methylene impurity (desferrioxamine A_1 and/or other desferrioxamines).

[b] All des-methylene impurities that elute in the 0.85–0.87 range should be treated as a single impurity. Where the cluster of unresolved peaks in this range is present, it should be integrated together as one peak to determine compliance.

[c] The acceptance criterion of NMT 3.0% applies to the sum of the peaks in the specified range.

SPECIFIC TESTS

• **pH** ⟨791⟩**:** 4.0–6.0, in a solution (1 in 100)

• **Water Determination,** *Method I*⟨921⟩**:** NMT 2.0%

• **Sterility Tests** ⟨71⟩**:** Where the label states that Deferoxamine Mesylate is sterile, it meets the requirements.

• **Bacterial Endotoxins Test** ⟨85⟩**:** Where the label states that Deferoxamine Mesylate is sterile or must be subjected to further processing during the preparation of injectable dosage forms, it contains NMT 0.33 USP Endotoxin Unit/mg of deferoxamine mesylate.

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight containers. Store at room temperature.

• **Labeling:** Where it is intended for use in preparing injectable dosage forms, the label states that it is sterile or must be subjected to further processing during the preparation of injectable dosage forms.

• **USP Reference Standards** ⟨11⟩

USP Deferoxamine Mesylate RS

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

Topic/Question	Contact	Expert Committee
DEFEROXAMINE MESYLATE	Documentary Standards Support	SM32020 Small Molecules 3

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. PF 39(1)

Current DocID: GUID-2DA07CF4-1552-473A-BC94-B87C1B7C97B5_4_en-US

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