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Add the following:

▲Gemifloxacin Tablets

DEFINITION

Gemifloxacin Tablets contain an amount of gemifloxacin mesylate equivalent to NLT 90.0% and NMT 110.0% of the labeled amount of gemifloxacin ($C_{18}H_{20}FN_5O_4$).

IDENTIFICATION

• **A.** **Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*:** 197A or 197M

• **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**

Store the solutions in amber vials and protect from light.

Buffer: 138 g/L of monobasic sodium phosphate in water

Mobile phase: Acetonitrile, water, and trifluoroacetic acid (20: 80: 0.1)

Diluent: Acetonitrile, water, and *Buffer* (20:72:8)

Standard solution: 0.17 mg/mL of USP Gemifloxacin Mesylate RS in *Diluent*

Sample stock solution: Nominally 0.64 mg/mL of gemifloxacin from a portion of finely powdered Tablets in *Diluent* prepared as follows. Finely powder NLT 20 Tablets. Transfer a suitable portion of the powder to a suitable volumetric flask. Add *Diluent* using 70% of the final volume, shake it for 15 min, and then sonicate for about 30 min. Allow the solution to cool, if necessary, and dilute with *Diluent* to volume.

Sample solution: Nominally 0.13 mg/mL of gemifloxacin from the *Sample stock solution* in *Diluent*

Chromatographic system

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

Mode: LC

Detector: UV 272 nm

Column: 4.6-mm × 25-cm; 5-µm packing L1

Column temperature: 30°

Flow rate: 1 mL/min

Injection volume: 20 µL

Run time: NLT 1.3 times the retention time of the gemifloxacin peak

System suitability

Sample: *Standard solution*

Suitability requirements

Tailing factor: NMT 2.0

Relative standard deviation: NMT 1.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of gemifloxacin ($C_{18}H_{20}FN_5O_4$) in the portion of Tablets taken:

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

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

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

C_S = concentration of USP Gemifloxacin Mesylate RS in the *Standard solution* (mg/mL)

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

M_{r1} = molecular weight of gemifloxacin, 389.38

M_{r2} = molecular weight of gemifloxacin mesylate, 485.49

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

• **Dissolution ⟨711⟩**

Medium: 0.01 N hydrochloric acid; 900 mL

Apparatus 2: 50 rpm

Time: 30 min

Standard solution: 0.01 mg/mL of USP Gemifloxacin Mesylate RS in *Medium*

Sample solution: Pass a portion of the solution under test through a suitable filter of 0.45-µm pore size, and dilute with *Medium* to a concentration that is similar to the *Standard solution*.

Instrumental conditions

Mode: UV

Detector: 343 nm

Blank: *Medium*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of gemifloxacin ($C_{18}H_{20}FN_5O_4$) dissolved:

$$\text{Result} = (A_U/A_S) \times (C_S/L) \times (M_{r1}/M_{r2}) \times D \times V \times 100$$

A_U = absorbance of the *Sample solution*

A_S = absorbance of the *Standard solution*

C_S = concentration of USP Gemifloxacin Mesylate RS in the *Standard solution* (mg/mL)

L = label claim (mg/Tablet)

M_{r1} = molecular weight of gemifloxacin, 389.38

M_{r2} = molecular weight of gemifloxacin mesylate, 485.49

D = dilution factor of the *Sample solution*, as needed

V = volume of *Medium*, 900 mL

Tolerances: NLT 70% (Q) of the labeled amount of gemifloxacin ($C_{18}H_{20}FN_5O_4$) is dissolved.

• **Uniformity of dosage units ⟨905⟩:** Meet the requirements

IMPURITIES

Store the solutions in amber vials and protect from light.

• **Organic Impurities**

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

System suitability solution: 0.5 mg/mL of USP Gemifloxacin Mesylate RS in *Diluent*

Sensitivity solution: 0.06 µg/mL of USP Gemifloxacin Mesylate RS in *Diluent*

System suitability

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

Suitability requirements

Resolution: NLT 5.0 between *E*-gemifloxacin and gemifloxacin, *System suitability solution*

Tailing factor: NMT 2.0, *Standard solution*

Relative standard deviation: NMT 1.0%, *Standard solution*

Signal-to-noise ratio: NLT 10, *Sensitivity solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of any individual impurity in the portion of Tablets taken:

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

r_U = peak response of any individual impurity from the *Sample solution*

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

C_S = concentration of USP Gemifloxacin Mesylate RS in the *Standard solution* (mg/mL)

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

M_{r1} = molecular weight of gemifloxacin, 389.38

M_{r2} = molecular weight of gemifloxacin mesylate, 485.49

Acceptance criteria: See *Table 1*. The reporting threshold is 0.1%.

Table 1

Name	Relative Retention Time	Acceptance Criteria, NMT (%)
E-Gemifloxacin[a]	0.78	1.0
Gemifloxacin	1.0	—
Any other individual impurity	—	0.2
Total impurities	—	1.5

[a] (*E*)-7-[3-(Aminomethyl)-4-(methoxyimino)pyrrolidin-1-yl]-1-cyclopropyl-6-fluoro-4-oxo-1,4-dihydro-1,8-naphthyridine-3-carboxylic acid monomethanesulfonate.

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in tightly closed containers, protected from light. Store at controlled room temperature.
- **USP Reference Standards ⟨11⟩**

USP Gemifloxacin Mesylate RS▲ (USP 1-May-2022)

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

Topic/Question	Contact	Expert Committee
GEMIFLOXACIN TABLETS	Documentary Standards Support	SM12020 Small Molecules 1

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
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