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Add the following:

▲Gefitinib Tablets

DEFINITION
Gefitinib Tablets contain NLT 90.0% and NMT 110.0% of the labeled amount of gefitinib ($C_{22}H_{24}ClFN_4O_3$).

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

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

ASSAY
• **Procedure**

Solution A: 9.7 g/L of ammonium acetate in water

Solution B: 0.2% trifluoroacetic acid in water

Mobile phase: Acetonitrile and *Solution A* (38:62)

Diluent: Acetonitrile and *Solution B* (40:60)

System suitability solution: 0.35 mg/mL of USP Gefitinib RS and 0.25 mg/mL of USP Dichloroaniline RS in *Diluent*. Sonicate to dissolve.

Standard solution: 0.35 mg/mL of USP Gefitinib RS in *Diluent*. Sonicate to dissolve.

Sample solution: Nominally 0.35 mg/mL of gefitinib prepared as follows. Transfer a suitable amount of the fine powder, equivalent to about 35 mg of gefitinib, from NLT 10 Tablets to a 100-mL volumetric flask. Add about 70 mL of *Diluent* and sonicate to dissolve. Dilute with *Diluent* to volume. Pass a portion of the solution through a suitable filter of 0.45-µm pore size. Discard the first few milliliters of filtrate.

Chromatographic system

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

Mode: LC

Detector: UV 247 nm. For *Identification B*, use a diode array detector in the range of 200–400 nm.

Column: 3.0-mm × 10-cm; 3-µm packing L1

Column temperature: 60°

Flow rate: 0.9 mL/min

Injection volume: 5 µL

Run time: NLT 5 times the retention time of gefitinib

System suitability

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

Suitability requirements

Resolution: NLT 5.0 between dichloroaniline and gefitinib, *System suitability solution*

Tailing factor: NMT 1.5, *Standard solution*

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

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of gefitinib ($C_{22}H_{24}ClFN_4O_3$) in the portion of Tablets taken:

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

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

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

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

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

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

• **Dissolution ⟨711⟩**

Medium: 5% polysorbate 80 in water; 1000 mL

Apparatus 2: 50 rpm

Time: 45 min

Standard solution: 0.25 mg/mL of USP Gefitinib RS in *Medium*

Sample solution: Pass a portion of the solution under test through a suitable filter of 0.45-µm pore size, discarding the first few milliliters.

Blank: *Medium*

Instrumental conditions

(See *Ultraviolet-Visible Spectroscopy ⟨857⟩*.)

Mode: UV

Analytical wavelength: 334 nm

Cell: 1 mm

Analysis:

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of gefitinib ($C_{22}H_{24}ClFN_4O_3$) dissolved:

$$\text{Result} = (A_U/A_S) \times C_S \times V \times (1/L) \times 100$$

A_U = absorbance of the *Sample solution*

A_S = absorbance of the *Standard solution*

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

V = volume of *Medium*, 1000 mL

L = label claim (mg/Tablet)

Tolerances: NLT 80% (Q) of the labeled amount of gefitinib ($C_{22}H_{24}ClFN_4O_3$) is dissolved.

• **Uniformity of Dosage Units ⟨905⟩:** Meet the requirements

IMPURITIES

• **Organic Impurities**

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

Standard stock solution: Use the *Standard solution* as prepared in the *Assay*.

Standard solution: 0.7 µg/mL of USP Gefitinib RS from the *Standard stock solution* in *Diluent*

Sensitivity solution: 0.18 µg/mL of USP Gefitinib RS from the *Standard solution* in *Diluent*

System suitability

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

[Note—The relative retention times for dichloroaniline and gefitinib are 0.7 and 1.0, respectively. These relative retention times are provided as information that could aid in peak assignment.]

Suitability requirements

Resolution: NLT 5.0 between dichloroaniline and gefitinib, *System suitability solution*

Relative standard deviation: NMT 5.0%, *Standard solution*

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

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of any unspecified degradation product in the portion of Tablets taken:

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

r_U = peak response of any unspecified degradation product from the *Sample solution*

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

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

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

Acceptance criteria: See *Table 1*. The reporting threshold is 0.05%.

Table 1

Name	Acceptance Criteria, NMT (%)
Any unspecified degradation product	0.2
Total degradation products	0.4

ADDITIONAL REQUIREMENTS

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

• **USP Reference Standards** ⟨11⟩

USP Dichloroaniline RS

3,4-Dichloroaniline.

$C_6H_5Cl_2N$ 162.01

USP Gefitinib RS▲ (USP 1-Aug-2024)

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

Topic/Question	Contact	Expert Committee
GEFITINIB TABLETS	Documentary Standards Support	SM32020 Small Molecules 3

Chromatographic Database Information: Chromatographic Database

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