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Piroxicam Capsules

DEFINITION

Piroxicam Capsules contain NLT 92.5% and NMT 107.5% of the labeled amount of piroxicam ($C_{15}H_{13}N_3O_4S$).

IDENTIFICATION

• **A. Thin-Layer Chromatography**

Diluent: Chloroform and methanol (1:1)

Standard solution: 1 mg/mL of USP Piroxicam RS in *Diluent*

Sample solution: Dissolve a portion of the contents of Capsules in *Diluent* to obtain a solution containing about 1 mg/mL. Shake by mechanical means for 10 min, and filter a portion. Use the filtrate for the analysis.

Chromatographic system

(See *Chromatography ⟨621⟩, Thin-Layer Chromatography*.)

Absorbent: 0.25-mm layer of chromatographic silica gel

Application volume: 20 µL

Developing solvent system: Toluene and glacial acetic acid (95:5)

Analysis

Samples: *Standard solution* and *Sample solution*

Allow the spots to dry, and develop the chromatogram in the *Developing solvent system* until the solvent front has moved about three-fourths of the length of the plate. Remove the plate from the developing chamber, and air-dry. Place the plate in the developing chamber, and develop as before. Remove the plate from the chamber, mark the solvent front, and air-dry. Locate the spots on the plate by viewing under short-wavelength UV light.

Acceptance criteria: The R_F value of the principal spot of the *Sample solution* corresponds to that of the *Standard solution*.

ASSAY

• **Procedure**

Solution A: Dissolve 7.72 g of anhydrous citric acid in 400 mL of water.

Solution B: Dissolve 5.35 g of dibasic sodium phosphate in 100 mL of water.

Solution C: Add *Solution B* to *Solution A*, and dilute with water to make 1000 mL.

Mobile phase: Methanol and *Solution C* (450:550)

Diluent: 0.01 N methanolic hydrochloric acid

Standard stock solution: 0.25 mg/mL of USP Piroxicam RS in *Diluent*

Standard solution: 0.05 mg/mL of USP Piroxicam RS prepared as follows. Transfer 10.0 mL of *Standard stock solution* to a 50-mL volumetric flask, add 25 mL of *Diluent* and 10.0 mL of water, and dilute with *Diluent* to volume.

Sample stock solution: Transfer, as completely as possible, the contents of NLT 20 Capsules to a suitable tared container, and determine the average weight per capsule. Mix the combined contents, and transfer an amount nominally equivalent to about 50 mg of piroxicam to a 100-mL volumetric flask. Add about 70 mL of *Diluent*, and shake by mechanical means for 30 min. Dilute with *Diluent* to volume, and mix. Centrifuge a portion of this mixture to obtain a clear solution.

Sample solution: Transfer 10.0 mL of the *Sample stock solution* to a 100-mL volumetric flask, add about 50 mL of *Diluent* and 20.0 mL of water, dilute with *Diluent* to volume, and mix.

Chromatographic system

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

Mode: LC

Detector: UV 254 nm

Column: 3.9-mm × 30-cm; packing L1

Flow rate: 1.2 mL/min

Injection volume: 25 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Column efficiency: NLT 500 theoretical plates

Tailing factor: NMT 1.5

Relative standard deviation: NMT 2.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of piroxicam ($C_{15}H_{13}N_3O_4S$) in the portion of Capsules taken:

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

r_U = peak response from the *Sample solution*

r_S = peak response from the *Standard solution*

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

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

Acceptance criteria: 92.5%–107.5%

PERFORMANCE TESTS

• **Dissolution ⟨711⟩**

Medium: Simulated gastric fluid TS, prepared without pepsin; 900 mL

Apparatus 1: 50 rpm

Time: 45 min

Standard stock solution: 0.5 mg/mL of USP Piroxicam RS in methanol

Standard solution: A known concentration of USP Piroxicam RS in *Medium* from *Standard stock solution*. Dilute with *Medium*, if necessary.

Sample solution: Filter a portion of the solution under test, suitably dilute with *Medium*, if necessary.

[Note—Use a suitable filter that does not adsorb piroxicam.]

Instrumental conditions

Mode: UV

Analytical wavelength: Maximum at about 333 nm

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of piroxicam ($C_{15}H_{13}N_3O_4S$) dissolved by comparing the UV absorbance of *Sample solution* with that of *Standard solution*.

Tolerances: NLT 75% (*Q*) of the labeled amount of piroxicam ($C_{15}H_{13}N_3O_4S$) is dissolved.

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

SPECIFIC TESTS

• **Water Determination, *Method I*⟨921⟩:** NMT 8.0%

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight, light-resistant containers.

• **USP Reference Standards ⟨11⟩**

USP Piroxicam RS

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

Topic/Question	Contact	Expert Committee
PIROXICAM CAPSULES	Documentary Standards Support	SM22020 Small Molecules 2
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM22020 Small Molecules 2

Chromatographic Database Information: Chromatographic Database

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