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Raloxifene Hydrochloride Tablets

DEFINITION

Raloxifene Hydrochloride Tablets contain NLT 93.0% and NMT 107.0% of the labeled amount of raloxifene hydrochloride ($C_{28}H_{27}NO_4S \cdot HCl$).

IDENTIFICATION

Change to read:

- **A.** ▲ [SPECTROSCOPIC IDENTIFICATION TESTS \(197\)](#), [Infrared Spectroscopy: 197K](#)▲ (CN 1-MAY-2020)

Sample: Transfer a quantity of powdered Tablets, equivalent to 120 mg of raloxifene hydrochloride, to a suitable container. Add 20 mL of [water](#), and shake to form a uniform slurry. Centrifuge, and discard the supernatant. Add 5 mL of [isopropyl alcohol](#), shake to form a slurry, filter, and rinse the residue with [isopropyl alcohol](#). Dry the residue at 105° for 30 min. Alternatively, add 40 mL of [water](#) to the powdered Tablets and vortex to form a uniform slurry. Centrifuge, and discard the supernatant. Repeat the washing process. Add 40 mL of [methanol](#) to the residue, vortex to form a uniform slurry, and centrifuge. Transfer the clear liquid to an appropriate container and evaporate to dryness.

Analysis: Prepare a potassium bromide dispersion with the *Sample*. Similarly prepare the *Standard*, starting with a slurry containing 12 mg/mL of [USP Raloxifene Hydrochloride RS](#) in water.

Acceptance criteria: Meet the requirements

- **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

Buffer: Dissolve 7.2 g of [monobasic potassium phosphate](#) in 1000 mL of [water](#). Add 1.3 mL of [phosphoric acid](#), and further adjust with [phosphoric acid](#) or [potassium hydroxide](#) solution to a pH of 2.5 ± 0.1.

Mobile phase: Acetonitrile and *Buffer* (33:67)

Diluent: Acetonitrile and *Buffer* (60:40)

System suitability solution: Prepare as directed in the test for *Organic Impurities*.

Standard solution: 0.06 mg/mL of [USP Raloxifene Hydrochloride RS](#) in *Diluent*

Sample solution: Transfer a sufficient quantity of Tablets to a volumetric flask of suitable size, add *Diluent*, and shake to disintegrate the Tablets. Sonicate if necessary. Dilute with *Diluent* to obtain a solution having a concentration of 0.06 mg/mL of raloxifene hydrochloride, based on the label claim. Filter, and use the clear solution.

Chromatographic system

(See [Chromatography \(621\)](#), [System Suitability](#).)

Mode: LC

Detector: UV 280 nm

Column: 4.6-mm × 15-cm; 3.5-μm base-deactivated packing [L7](#)

Column temperature: 35°

Flow rate: 1.5 mL/min

Injection volume: 10 μL

System suitability

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

Suitability requirements

Resolution: NLT 2.0 between raloxifene and raloxifene related compound C, *System suitability solution*

Tailing factor: NMT 2.0 for raloxifene, *System suitability solution*

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

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of raloxifene hydrochloride ($C_{28}H_{27}NO_4S \cdot HCl$) in the portion of Tablets 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 Raloxifene Hydrochloride RS](#) in the *Standard solution* (mg/mL)

C_U = nominal concentration of raloxifene hydrochloride from the *Sample solution* (mg/mL)

Acceptance criteria: 93.0%–107.0%

PERFORMANCE TESTS

• [DISSOLUTION \(711\)](#)

Test 1

Medium: 0.1% [polysorbate 80](#); 1000 mL

Apparatus 2: 50 rpm

Time: 30 min

Mobile phase: Acetonitrile, [water](#), and [triethylamine](#) (500:500:2). Adjust with [phosphoric acid](#) to a pH of 4.0.

Triethylamine phosphate suspension: Add 2.0 mL of [triethylamine](#) to 500 mL of acetonitrile, and adjust with [phosphoric acid](#) to a pH of 4.0.

[NOTE—Triethylamine phosphate will precipitate; keep the suspension well mixed.]

Standard solution: Prepare a solution having a known concentration equivalent to the expected concentration of the *Sample solution* by dissolving [USP Raloxifene Hydrochloride RS](#) in a small volume (NMT 10% of the final volume) of [methanol](#). Dilute with *Medium* to volume, and mix the resulting solution with *Triethylamine phosphate suspension* (1:1).

Sample solution: Pass a portion of the solution under test through an appropriate filter of 0.45- μ m pore size. Mix the resulting solution and *Triethylamine phosphate suspension* (1:1).

Chromatographic system

(See [Chromatography \(621\)](#), [System Suitability](#).)

Mode: LC

Detector: UV 290 nm

Column: 4.6-mm $\times$ 15-cm; 3.5- μ m base-deactivated packing [L10](#). If the analyte peak splits, use a guard column containing packing [L3](#).

Flow rate: 2 mL/min

Injection volume: 50 μ L

System suitability

Sample: *Standard solution*

Suitability requirements

Relative standard deviation: NMT 2.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Determine the percentage of the labeled amount of raloxifene hydrochloride ($C_{28}H_{27}NO_4S \cdot HCl$) dissolved:

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

r_U = peak response from the *Sample solution*

r_S = peak response from the *Standard solution*

C_S = concentration of [USP Raloxifene Hydrochloride RS](#) in the *Standard solution* (mg/mL)

L = label claim (mg/Tablet)

F = volume of *Medium*, 1000 mL

Tolerances: NLT 80% (Q) of the labeled amount of raloxifene hydrochloride is dissolved.

Test 2: If the product complies with this test, the labeling indicates that it meets USP *Dissolution Test 2*.

Medium: 1% [sodium dodecyl sulfate](#) in 0.05 M phosphate buffer prepared as follows. 1.7 g/L of [sodium hydroxide](#) and 7 g/L of [monobasic sodium phosphate monohydrate](#). Adjust to a pH of 7.5, if necessary. Add 10 g of [sodium dodecyl sulfate](#) per L; 9.0 g/L.

Apparatus 2: 75 rpm