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Diltiazem Hydrochloride Tablets

DEFINITION

Diltiazem Hydrochloride Tablets contain NLT 90.0% and NMT 110.0% of the labeled amount of diltiazem hydrochloride ($C_{22}H_{26}N_2O_4S \cdot HCl$).

IDENTIFICATION

- **A.** The UV-Vis spectrum of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the Assay.
- **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: 0.79 g/L of ammonium bicarbonate in water. Adjust with diluted ammonia solution to a pH of 8.0.

Solution B: Acetonitrile

Mobile phase: See [Table 1](#).

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	95	5
2.0	95	5
5.0	60	40
13.0	60	40
16.0	30	70
20.0	30	70
20.1	95	5
25.0	95	5

Diluent: Acetonitrile and water (40:60)

Standard solution: 0.05 mg/mL of [USP Diltiazem Hydrochloride RS](#) in *Diluent*

Sample stock solution: Nominally 0.5 mg/mL of diltiazem hydrochloride from Tablets in *Diluent* prepared as follows. Transfer an appropriate portion of finely powdered Tablets (NLT 20) to a suitable volumetric flask. Add *Diluent* equivalent to 80% of the flask volume, and sonicate for 60 min. Dilute with *Diluent* to volume. Centrifuge the solution for 20 min. Use the supernatant.

Sample solution: Nominally 0.05 mg/mL of diltiazem hydrochloride in *Diluent* from *Sample stock solution*

Chromatographic system

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

Mode: LC

Detector: UV 240 nm. For *Identification* test A, use a diode-array detector in the range of 190–400 nm.

Column: 2.1-mm × 15-cm; 1.7-μm packing L1

Flow rate: 0.3 mL/min

Injection volume: 2.0 μL

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 diltiazem hydrochloride ($C_{22}H_{26}N_2O_4S \cdot HCl$) in the portion of Tablets taken:

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

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

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

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

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

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

• [DISSOLUTION \(711\)](#)

Medium: Water; 900 mL

Apparatus 2: 75 rpm

Time: 30 min and 3 h

Detector: UV 237 nm

Standard solution: [USP Diltiazem Hydrochloride RS](#) in *Medium*

Sample solution: Sample per [Dissolution \(711\)](#). Dilute with *Medium* to a concentration that is similar to the *Standard solution*.

Tolerances: See [Table 2](#) for the 30-min time point. Use the criteria in [Dissolution \(711\)](#), *Acceptance Table 1* for the 3-h time point. NMT 60% (Q) of the labeled amount of diltiazem hydrochloride ($C_{22}H_{26}N_2O_4S \cdot HCl$) is dissolved in 30 min, and NLT 75% (Q) is dissolved in 3 h.

Table 2

Stage	Acceptance Criteria
S_1	No unit is more than Q.
S_2	Average value is equal to or less than Q, and no unit is greater than Q + 10%.
S_3	Average value is equal to or less than Q, and NMT 2 units are more than Q + 10%, and no unit is more than Q + 25%.

• [UNIFORMITY OF DOSAGE UNITS \(905\)](#): Meet the requirements

IMPURITIES

• ORGANIC IMPURITIES

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

Standard solution: 2.5 µg/mL each of [USP Desacetyl Diltiazem Hydrochloride RS](#) and [USP Diltiazem Hydrochloride RS](#) in *Diluent*

Sample solution: Nominally 0.5 mg/mL of diltiazem hydrochloride from Tablets in *Diluent* prepared as follows. Transfer an appropriate portion of the powdered Tablets (NLT 20) to a suitable volumetric flask. Add *Diluent* equivalent to 80% of the flask volume, and sonicate for 60 min. Dilute with *Diluent* to volume. Centrifuge the solution for 20 min. Use the supernatant.

System suitability

Sample: *Standard solution*

[NOTE—See [Table 3](#) for the relative retention times.]

Suitability requirements

Resolution: NLT 2.0 between desacetyl diltiazem and diltiazem

Relative standard deviation: NMT 3.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of desacetyl diltiazem in the portion of Tablets taken:

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

r_U = peak response of desacetyl diltiazem from the *Sample solution*

r_S = peak response of desacetyl diltiazem from the *Standard solution*

C_S = concentration of [USP Desacetyl Diltiazem Hydrochloride RS](#) in the *Standard solution* (µg/mL)