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Oxybutynin Chloride Extended-Release Tablets

DEFINITION

Oxybutynin Chloride Extended-Release Tablets contain NLT 90.0% and NMT 110.0% of the labeled amount of oxybutynin chloride ($C_{22}H_{31}NO_3 \cdot HCl$).

IDENTIFICATION

Change to read:

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

Standard: Dissolve 15 mg of [USP Oxybutynin Chloride RS](#) in 5 mL of [water](#). Adjust with [0.1 N sodium hydroxide](#) to a pH of between 7 and 8. Extract the solution twice with 10 mL of [ether](#). Combine the extracts, evaporate the ether, and dry under vacuum over [silica gel](#) for at least 30 min. Redissolve the dried residue in a small amount of [acetone](#), transfer the solution to an IR salt plate, and evaporate to cast a thin film.

Sample: Add a quantity of finely powdered Tablets, equivalent to about 15 mg of oxybutynin chloride, to 5 mL of [water](#) per Tablet. Mix for 1 min. Adjust with [0.1 N sodium hydroxide](#) to a pH between 7 and 8. Extract the solution twice with 10 mL of ether. Combine the extracts, evaporate the [ether](#), and dry under vacuum over [silica gel](#) for at least 30 min. Redissolve the dried residue in a small amount of [acetone](#), transfer the solution to an IR salt plate, and evaporate to cast a thin film.

- **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 1

Diluent: Use [water](#) adjusted with [phosphoric acid](#) to a pH of 3.5.

Solution A: [Methanol](#) and [acetonitrile](#) (1:1)

Mobile phase: [Acetonitrile](#), [triethylamine](#), and [water](#) (700:3:1300). Adjust with [phosphoric acid](#) to a pH of 3.9.

Impurity stock solution: 0.11 mg/mL of [USP Oxybutynin Related Compound A RS](#) in [acetonitrile](#)

Standard stock solution: 0.37 mg/mL of [USP Oxybutynin Chloride RS](#) in [acetonitrile](#)

System suitability solution: Transfer 10 mL of the *Standard stock solution* and 1 mL of the *Impurity stock solution* to a 100-mL volumetric flask, and dilute with *Diluent* to volume.

Standard solution: 0.1 mg/mL of [USP Oxybutynin Chloride RS](#) in *Diluent* from the *Standard stock solution*

Sample solution

For Tablets that contain 5 mg of oxybutynin chloride: Place 10 Tablets in a 500-mL volumetric flask, add 150 mL of *Solution A*, and stir for at least 4 h or until dissolved. Dilute with *Diluent* to volume. Mix thoroughly, centrifuge, and use the clear supernatant.

For Tablets that contain 10 mg or more of oxybutynin chloride: Place 10 Tablets in a 1000-mL volumetric flask, add 300 mL of *Solution A*, and stir for at least 4 h or until dissolved. Dilute with *Diluent* to volume. If necessary, make a further dilution with *Diluent* to obtain a solution having a final concentration equivalent to 0.1 mg/mL of oxybutynin chloride. Mix thoroughly, centrifuge, and use the clear supernatant.

Chromatographic system

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

Mode: LC

Detector: UV 220 nm

Column: 4.6-mm × 15-cm; packing L11

Flow rate: 1.5 mL/min

Injection volume: 50 µL

System suitability

Sample: *System suitability solution*

[NOTE—The relative retention times for oxybutynin and oxybutynin related compound A are about 1.0 and 1.6, respectively.]

Suitability requirements

Resolution: NLT 1.5 between oxybutynin and oxybutynin related compound A

Tailing factor: Greater than 0.75 and NMT 2.5 for each peak

Relative standard deviation: NMT 3% for each compound for six replicate injections

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of oxybutynin chloride ($C_{22}H_{31}NO_3 \cdot HCl$) 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 Oxybutynin Chloride RS](#) in the *Standard solution* (mg/mL)

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

Acceptance criteria: 90.0%–110.0%

- **PROCEDURE 2:** Use *Procedure 2* for Tablets labeled to meet the requirements of USP *Dissolution Test 9*.

Mobile phase, Chromatographic system, System suitability and Analysis: Proceed as directed in *Assay Procedure 1*.

Diluent: [Methanol](#) and [water](#) (80:20)

Impurity stock solution: 0.11 mg/mL of [USP Oxybutynin Related Compound A RS](#) in [methanol](#). Sonicate to dissolve, if necessary.

Standard stock solution: 0.37 mg/mL of [USP Oxybutynin Chloride RS](#) in *Diluent*. Sonicate to dissolve, if necessary.

System suitability solution: Transfer 10 mL of the *Standard stock solution* and 1 mL of the *Impurity stock solution* to a 100-mL volumetric flask, and dilute with *Diluent* to volume.

Standard solution: 0.1 mg/mL of [USP Oxybutynin Chloride RS](#) in *Diluent* from the *Standard stock solution*

Sample solution: Nominally 0.1 mg/mL of oxybutynin chloride prepared as follows. Place 10 Tablets in an appropriate volumetric flask, add 60% of the flask volume of *Diluent*, and sonicate for at least 60 min with intermittent shaking. Maintain the temperature of the sonicator between 20 and 25°. Dilute with *Diluent* to volume. Mix thoroughly, centrifuge, and use the clear supernatant. Further dilute with *Diluent* as needed. [NOTE—Centrifuging at 6000 rpm for 10 min may be suitable.]

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

Change to read:

- [DISSOLUTION \(711\)](#).

Test 1

Medium: [Simulated gastric fluid](#) without enzyme; 50 mL

Apparatus 7: See [Drug Release \(724\)](#), 30 cycles/min; 2–3-cm amplitude, at $37.0 \pm 0.5^\circ$

Times: 4, 10, and 24 h

Solution A: 4.83 g/L of [monobasic sodium phosphate](#) in [water](#). Add 2.3 mL/L of [triethylamine](#), and adjust with [phosphoric acid](#) to a pH of 2.2 ± 0.2 .

Mobile phase: [Acetonitrile](#) and *Solution A* (7:13)

Solution B: To 1 L of [water](#) add [phosphoric acid](#) dropwise to a pH of 3.5, and mix well.

Standard stock solutions: 250, 300, and 350 µg/mL of [USP Oxybutynin Chloride RS](#) in [acetonitrile](#)

Standard solutions: Prepare a series of dilutions of the *Standard stock solutions* in *Solution B* having final concentrations similar to those expected in the *Sample solution*.

System suitability solution: Use a medium range *Standard solution* of [USP Oxybutynin Chloride RS](#).

Sample solution: Use portions of the solution under test. If the solution is cloudy, centrifuge at 2000 rpm for 10 min, and use the supernatant.

Chromatographic system

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

Mode: LC

Detector: UV 230 nm

Column: 4.6-mm × 5-cm; packing L11

Column temperature: 35°

Flow rate: 1.5 mL/min

Injection volume: 50 µL

System suitability**Sample:** *System suitability solution***Suitability requirements****Tailing factor:** Greater than 0.5 and less than 2.5**Relative standard deviation:** NMT 2.0%**Analysis****Samples:** *Standard solutions* and *Sample solution*

Construct a calibration curve by plotting the peak response versus concentration of the *Standard solutions*. A weighing factor, $1/x$, is applied to the regression line of the calibration curve to enhance the accuracy of the low standard concentrations.

Determine the percentage of oxybutynin chloride ($C_{22}H_{31}NO_3 \cdot HCl$) dissolved in each interval from a linear regression analysis of the calibration curve.

Tolerances: See [Tables 1](#) and [2](#).**Table 1. For Tablets Labeled to Contain 5 or 10 mg of Oxybutynin Chloride**

Time (h)	Amount Dissolved
4	NMT 20%
10	34.5%–59.5%
24	NLT 80%

Table 2. For Tablets Labeled to Contain 15 mg of Oxybutynin Chloride

Time (h)	Amount Dissolved
4	NMT 20%
10	34.5%–59.5%
24	NLT 75%

The percentages of the labeled amount of oxybutynin chloride ($C_{22}H_{31}NO_3 \cdot HCl$) dissolved at the times specified conform to [Dissolution \(711\)](#), [Acceptance Table 2](#).

Test 2: If the product complies with this test, the labeling indicates that the product meets USP *Dissolution Test 2*.**Acid stage medium:** [Simulated gastric fluid](#), without enzymes, pH 1.2 ± 0.05 ; 250 mL (first row)**Buffer stage medium:** [Simulated !\[\]\(5d954b3e270654ad8ab0d5913161c03c_img.jpg\) intestinal !\[\]\(b221a91d92ddcb14c8313ca57b8e9677_img.jpg\) \(ERR-1-May-2020\) fluid](#), without enzymes, pH 6.8 ± 0.1 ; 250 mL (rows 2–4)**Apparatus 3:** 25 dips/min; 20-mesh polypropylene screen on top and bottom; 30 s drip time**Times:** 2 h in the *Acid stage medium* (first row); 4, 8, and 16 h (corresponding to 2, 6, and 14 h after changing the medium) in the *Buffer stage medium* (rows 2–4)**Solution A:** Transfer 1 mL of [triethylamine](#) to 1000 mL of [water](#). Adjust with [phosphoric acid](#) to a pH of 3.50 ± 0.05 .**Mobile phase:** [Acetonitrile](#) and *Solution A* (4:1)**Standard stock solution:** 0.2 mg/mL of [USP Oxybutynin Chloride RS](#) in *Acid stage medium***Working standard solution:** Transfer 5.0 mL of the *Standard stock solution* for Tablets labeled to contain 5 mg, transfer 10 mL for Tablets labeled to contain 10 mg, or transfer 15 mL for Tablets labeled to contain 15 mg to a 100-mL volumetric flask. Dilute with *Buffer stage medium* to volume.**Sample solution:** Centrifuge a portion of the solution under test at approximately 3000 rpm for 10 min. Use the supernatant.**Chromatographic system**(See [Chromatography \(621\)](#), [System Suitability](#).)**Mode:** LC**Detector:** UV 203 nm**Column:** 4.6-mm $\times$ 25-cm; packing L7**Flow rate:** 1.5 mL/min

Injection volume: 25 µL

System suitability

Sample: Working standard solution

Suitability requirements

Tailing factor: NMT 2.0

Relative standard deviation: NMT 3.0%

Analysis

Samples: Working standard solution and Sample solution

Calculate the percentage of the labeled amount of oxybutynin chloride ($C_{22}H_{31}NO_3 \cdot HCl$) dissolved at each time point ($C_{T2}, C_{T4}, C_{T8}, C_{T16}$):

$$C_i = (r_U/r_S) \times (C_S/L) \times V \times 100$$

r_U = peak response from the Sample solution

r_S = peak response from the Working standard solution

C_S = concentration of the Working standard solution (mg/mL)

L = label claim (mg/Tablet)

V = volume of Medium, 250 mL

C_{T2} = percentage dissolved at 2 h, C_2

C_{T4} = percentage dissolved at 4 h, $C_2 + C_4$

C_{T8} = percentage dissolved at 8 h, $C_2 + C_4 + C_8$

C_{T16} = percentage dissolved at 16 h, $C_2 + C_4 + C_8 + C_{16}$

Tolerances: See [Tables 3](#) and [4](#).

Table 3. For Tablets Labeled to Contain 5 or 10 mg of Oxybutynin Chloride

Time (h)	Amount Dissolved
2	0%–10%
4	10%–30%
8	40%–65%
16	NLT 80%

Table 4. For Tablets Labeled to Contain 15 mg of Oxybutynin Chloride

Time (h)	Amount Dissolved
2	0%–10%
4	10%–30%
8	35%–65%
16	NLT 75%

The percentages of the labeled amount of oxybutynin chloride ($C_{22}H_{31}NO_3 \cdot HCl$) dissolved at the times specified conform to [Dissolution <711>](#), [Acceptance Table 2](#).

Test 3: If the product complies with this test, the labeling indicates that the product meets USP *Dissolution* Test 3.

