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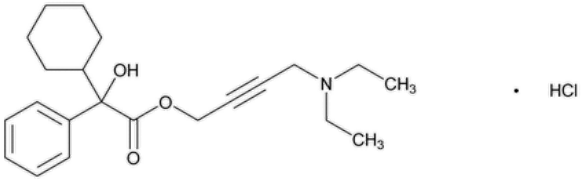
Oxybutynin Chloride



$C_{22}H_{31}NO_3 \cdot HCl$ 393.95

Benzeneacetic acid, α-cyclohexyl-α-hydroxy-, 4-(diethyl amino)-2-butynyl ester hydrochloride, (±)-;

4-(Diethylamino)-2-butynyl (±)-α-phenylcyclohexaneglycolate hydrochloride CAS RN®: 1508-65-2; UNII: L9F3D9RENQ.

DEFINITION

Oxybutynin Chloride contains NLT 97.0% and NMT 102.0% of oxybutynin chloride ($C_{22}H_{31}NO_3 \cdot HCl$), calculated on the dried basis.

IDENTIFICATION

Change to read:

• **A.** ▲**Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*: 197K**▲ (CN 1-May-2020)

• **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: A solution containing 6.67 g/L of monobasic potassium phosphate and 8.55 g/L of dibasic potassium phosphate

Mobile phase: Acetonitrile and *Buffer* (49:51)

Standard solution: 0.1 mg/mL of USP Oxybutynin Chloride RS in *Mobile phase*

Sample solution: 0.1 mg/mL of Oxybutynin Chloride in *Mobile phase*

Chromatographic system

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

Mode: LC

Detector: UV 210 nm

Column: 4.6-mm × 7.5-cm; 3-µm or 3.5-µm packing L7

Column temperature: 45°

Flow rate: 1 mL/min

Injection volume: 10 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Relative standard deviation: NMT 1.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of oxybutynin chloride ($C_{22}H_{31}NO_3 \cdot HCl$) in the portion of Oxybutynin Chloride 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 = concentration of Oxybutynin Chloride in the *Sample solution* (mg/mL)

Acceptance criteria: 97.0%–102.0% on the dried basis

OTHER COMPONENTS

• **Chloride Content**

Sample: 600 mg of Oxybutynin Chloride, previously dried

Analysis: Dissolve the *Sample* in 100 mL of water, and add 5 mL of nitric acid. Titrate (see *Titrimetry ⟨541⟩*) with 0.1 N silver nitrate VS, using a platinum–silver chloride electrode system. Each mL of 0.1 N silver nitrate is equivalent to 3.545 mg of chloride (Cl).

Acceptance criteria: 8%–10%

IMPURITIES

• **Residue on Ignition ⟨281⟩:** NMT 0.1%

• **Organic Impurities**

Buffer, Mobile phase, and **Chromatographic system:** Proceed as directed in the *Assay*.

System suitability stock solution: 100 µg/mL each of USP Oxybutynin Related Compound B RS and USP Oxybutynin Related Compound C RS in *Mobile phase*

Standard stock solution: 1.0 mg/mL of USP Oxybutynin Chloride RS in *Mobile phase*

System suitability solution: Transfer 10.0 mL of the *System suitability stock solution* to a 100-mL volumetric flask, add 10.0 mL of *Standard stock solution*, and dilute with *Mobile phase* to volume.

Standard solution: 7.5 µg/mL of USP Oxybutynin Chloride RS from *Standard stock solution* in *Mobile phase*

Sample solution: 5.0 mg/mL of Oxybutynin Chloride in *Mobile phase*

System suitability

Sample: *System suitability solution*

[Note—See *Table 1* for relative retention times.]

Suitability requirements

Resolution: NLT 1.1 between oxybutynin related compound B and oxybutynin related compound C

Relative standard deviation: NMT 2.0% for the oxybutynin peak

Analysis

Samples: *Standard solution* and *Sample solution*

Record the chromatograms for a total time of NLT twice the retention time of the oxybutynin peak.

Calculate the percentage of each impurity in the portion of Oxybutynin Chloride taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times (1/F) \times 100$$

r_U = peak response of each impurity from the *Sample solution*

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

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

C_U = concentration of Oxybutynin Chloride in the *Sample solution* (mg/mL)

F = relative response factor for each impurity (see *Table 1*)

Acceptance criteria: See *Table 1*.

Table 1

Name	Relative Retention Time	Relative Response Factor	Acceptance Criteria, NMT (%)
Oxybutynin related compound A[a]	0.08	1.4	0.5

Name	Relative Retention Time	Relative Response Factor	Acceptance Criteria, NMT (%)
Diphenyl analog of oxybutynin chloride[b]	0.37	2.7	0.1
Oxybutynin related compound B[c]	0.65	1.3	1.0
Oxybutynin related compound C[d]	0.79	1.0	1.0
Oxybutynin	1.0	—	—
Cyclohexenyl analog of oxybutynin chloride[e]	1.8	0.4	1.0
Ethylpropyl analog of oxybutynin chloride[f]	1.9	1.0	0.1
Any other individual impurity	—	1.0	0.1
Total impurities	—	—	1.0

[a] Phenylcyclohexylglycolic acid (cyclohexylmandelic acid, or CHMA).

[b] 4-(Diethylamino)but-2-ynyl 2-hydroxy-2,2-diphenylacetate.

[c] Methyl ester of phenylcyclohexylglycolic acid (methyl ester of cyclohexylmandelic acid, or CHMME).

[d] Methylethyl analog of oxybutynin chloride (4-(ethylmethylamino) but-2-ynyl (±)-2-cyclohexyl-2-hydroxy-2-phenylacetate).

[e] 4-(Diethylamino)but-2-ynyl (±)-2-(cyclohex-3-enyl)-2-cyclohexyl-2-hydroxyacetate.

[f] 4-(Ethylpropylamino)but-2-ynyl (±)-2-cyclohexyl-2-hydroxy-2-phenylacetate.

SPECIFIC TESTS

• **Loss on Drying ⟨731⟩**

Analysis: Dry a sample at 105° for 2 h.

Acceptance criteria: NMT 3%

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in well-closed containers.

• **USP Reference Standards ⟨11⟩**

USP Oxybutynin Chloride RS

USP Oxybutynin Related Compound B RS

Methyl ester of phenylcyclohexylglycolic acid, or CHMME (cyclohexyl mandelic acid methyl ester).

USP Oxybutynin Related Compound C RS

Methylethyl analog of oxybutynin chloride, or 4-(ethylmethylamino) but-2-ynyl (±) 2-cyclohexyl-2-hydroxy-2-phenylacetate hydrochloride.

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

Topic/Question	Contact	Expert Committee
OXYBUTYNIN CHLORIDE	Documentary Standards Support	SM32020 Small Molecules 3
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM32020 Small Molecules 3

Chromatographic Database Information: Chromatographic Database

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