Oxycodone Hydrochloride Oral Solution

DEFINITION
Oxycodone Hydrochloride Oral Solution contains NLT 90.0% and NMT 110.0% of the labeled amount of oxycodone hydrochloride (C₁₈H₂₁NO₄ ´ HCl).

IDENTIFICATION

A. Sample solution: Transfer an amount equivalent to 15 mg of oxycodone from Oral Solution to a separatory funnel, add 10 mL of 0.01 N hydrochloric acid, and extract with four 40-mL portions of chloroform, collecting the chloroform extracts in a second separator. Wash the combined chloroform extracts with 5 mL of 0.01 N hydrochloric acid, and discard the chloroform layer. Combine the acidic wash with the aqueous solution remaining in the first separator and adjust with 6 N ammonium hydroxide to a pH of 9.5 ± 0.5. Extract with one 50-mL and two 20-mL portions of chloroform, and filter the chloroform extracts through chloroform-washed cotton, collecting the filtrate in a 100-mL volumetric flask. Dilute with water-saturated chloroform to volume, and mix.

Standard solution: Prepare a solution using 12 mg of USP Oxycodone RS and 25 mL of 0.01 N hydrochloric acid, and proceed as directed above, beginning with "extract with four 40-mL portions of chloroform".

Acceptance criteria: The UV absorption spectrum of the Sample solution exhibits maxima and minima at the same wavelengths as that of the Standard solution, concomitantly measured.

B. THIN-LAYER CHROMATOGRAPHY

Standard solution: Evaporate 5 mL of the Standard solution obtained from Identification test A just to dryness. Dissolve the residue in 1.0 mL of chloroform.

Sample solution: Evaporate 5 mL of the Sample solution obtained from Identification test A just to dryness. Dissolve the residue in 1.0 mL of chloroform.

Chromatographic system
(See Chromatography (621), Thin-Layer Chromatography.)

Mode: TLC
Adsorbent: 0.25-mm layer of chromatographic silica gel mixture
Application volume: 20 µL
Developing solvent system: Acetone, toluene, ether, and ammonium hydroxide (6:4:1:0.3)

Analysis
Samples: Standard solution and Sample solution

Develop the plate until the solvent front has moved about three-fourths of the length of the plate, remove it, mark the solvent front, allow the solvent to evaporate, and spray with iodoplatinate TS.

Acceptance criteria: The principal spot from the Sample solution corresponds in color, size, and Rf value to that from the solution from the Standard solution, and no other spots are observed.

C. The retention time of the oxycodone peak of the Sample solution corresponds to that of the Standard solution, as obtained in the Assay.

ASSAY

Procedure
Mobile phase: Acetonitrile, 0.01 M sodium 1-hexanesulfonate, and glacial acetic acid (25:74:1). Adjust with 5 N sodium hydroxide to a pH of 3.5.

Standard solution: 0.045 mg/mL of USP Oxycodone RS in Mobile phase

Sample solution: Transfer an amount equivalent to 5 mg of oxycodone hydrochloride from Oral Solution to a 100-mL volumetric flask, dilute with Mobile phase to volume, and mix. Pass a portion of this mixture through a filter having a 0.5-µm or finer pore size, and use the clear filtrate as the Sample solution.

Chromatographic system
(See Chromatography (621), System Suitability.)

Mode: LC
Detector: UV 280 nm
Column: 4.6-mm x 15-cm; 5-µm packing L1
Flow rate: 1.7 mL/min
Injection volume: 10 µL

System suitability
Sample: Standard solution
Suitability requirements
Tailing factor: NMT 2.0
Relative standard deviation: NMT 2.0%

Analysis
Samples: Standard solution and Sample solution

Calculate the percentage of oxycodone hydrochloride (C₁₈H₂₁NO₄ ´ HCl) in each mL of Oral Solution taken:

\[ \text{Result} = \left( \frac{r_U}{r_S} \right) \times \left( \frac{C_U}{C_S} \right) \times \left( \frac{M_r}{M_u} \right) \times 100 \]

where:
- \( r_U \) = peak response from the Sample solution
- \( r_S \) = peak response from the Standard solution
- \( C_U \) = concentration of USP Oxycodone RS in the Standard solution (mg/mL)
- \( C_S \) = concentration of oxycodone hydrochloride in the Sample solution (mg/mL)
- \( M_r \) = molecular weight of oxycodone base, 315.37
- \( M_u \) = molecular weight of oxycodone hydrochloride, 351.82

Acceptance criteria: 90.0%–110.0%

OTHER COMPONENTS

• ALCOHOL DETERMINATION, Method II (611): If present, 85.0%–115.0% of the labeled amount of alcohol (C₂H₅OH), determined by the gas-liquid chromatographic method, using acetone as the internal standard

PERFORMANCE TESTS

• UNIFORMITY OF DOSAGE UNITS (905): Meets the requirements for oral solution packaged in single-unit containers.

• DELIVERABLE VOLUME (698): Meets the requirements for oral solution packaged in multiple-unit containers.

SPECIFIC TESTS

Change to read:

• pH (791): 1.4–4.6 (88 1-Feb-2015)

ADDITIONAL REQUIREMENTS

• PACKAGING AND STORAGE: Preserve in tight, light-resistant containers.

• USP REFERENCE STANDARDS (11)
USP Oxycodone RS