Oxycodone Hydrochloride Oral Solution

DEFINITION

Oxycodone Hydrochloride Oral Solution contains NLT 90.0% and NMT 110.0% of the labeled amount of oxycodone hydrochloride ($C_{18}H_{21}NO_4 \cdot HCl$).

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 R_F 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 × 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 ($C_{18}H_{21}NO_4 \cdot HCl$) in each mL of Oral Solution taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times (M_{r1}/M_{r2}) \times 100$$

  r_U = peak response from the *Sample solution*
  r_S = peak response from the *Standard solution*
  C_S = concentration of USP Oxycodone RS in the *Standard solution* (mg/mL)
  C_U = nominal concentration of oxycodone hydrochloride in the *Sample solution* (mg/mL)
  M_{r1} = molecular weight of oxycodone hydrochloride, 351.82
  M_{r2} = molecular weight of oxycodone base, 315.37

  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 (C_2H_5OH), 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• (RB 1-Feb-2015)

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in tight, light-resistant containers.
- **USP REFERENCE STANDARDS** ⟨**11**⟩
  USP Oxycodone RS