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Pentazocine and Naloxone Tablets

DEFINITION

Pentazocine and Naloxone Tablets contain amounts of Pentazocine Hydrochloride and Naloxone Hydrochloride equivalent to NLT 90.0% and NMT 110.0% of the labeled amounts of pentazocine ($C_{19}H_{27}NO$) and naloxone ($C_{19}H_{21}NO_4$).

IDENTIFICATION

• **A.**

Diluent: Chloroform and methanol (1:1)

Standard solution A: 5.0 mg/mL of USP Pentazocine RS in *Diluent*

Standard solution B: 1.3 mg/mL of USP Naloxone RS in *Diluent*

Sample solution A: Crush 1 Tablet in 10 mL of *Diluent*. Sonicate for about 2 min, and filter.

Sample solution B: Evaporate 5 mL of *Sample solution A* to dryness on a steam bath under a stream of nitrogen. Dissolve the residue in 0.2 mL of *Diluent*.

Chromatographic system

(See *Chromatography ⟨621⟩, Thin-Layer Chromatography*.)

Adsorbent: 0.25-mm layer of chromatographic silica gel mixture

Application volume: 10 µL, *Standard solution A* and *Sample solution A*; 5 µL, *Standard solution B* and *Sample solution B*

Developing solvent system: 1-Butanol, water, and glacial acetic acid (70:20:10)

Spray reagent: Folin-Ciocalteu Phenol TS followed by sodium hydroxide solution (1 in 10)

Analysis: Develop the chromatograms in the *Developing solvent system* until the solvent has moved about three-fourths of the length of the plate. Remove the plate from the chamber, mark the solvent front, and dry under a current of warm air. Spray the plate with *Spray reagent*.

Acceptance criteria: *Sample solution A* and *Sample solution B* exhibit spots having the same R_F values and approximately the same sizes and shapes as their respective Standard solutions.

ASSAY

• **Procedure**

Diluent: Methanol, water, and phosphoric acid (500:500:1)

Solution A: Prepare a filtered and degassed mixture by dissolving 675 mg of sodium 1-octanesulfonate and 426 mg of anhydrous dibasic sodium phosphate in 625 mL of water, and mix.

Mobile phase: Add 475 mL of methanol and 10 mL of phosphoric acid to *Solution A*.

Strong anion-exchange resin: Transfer 30 g of strong anion-exchange resin to a 250-mL beaker. Wash the resin with two 200-mL portions of water, decanting the water after each wash. Wash with two 200-mL portions of dilute glacial acetic acid (1 in 20), decanting the first wash, and filter with the aid of suction.

Standard stock solution: 0.2 mg/mL of USP Naloxone RS in *Diluent*

Standard solution: Transfer 100 mg of USP Pentazocine RS to a 50-mL volumetric flask. Dissolve in about 30 mL of *Diluent*. Add 5.0 mL of the *Standard stock solution*, and dilute with *Diluent* to volume.

Sample solution: Transfer an amount nominally equivalent to 100 mg of pentazocine from NLT 20 Tablets to a 100-mL volumetric flask, and add 50.0 mL of *Diluent*. Sonicate for 5 min, and shake intermittently for 15 min. Filter into a glass-stoppered conical flask. Add about 250 mg of *Strong anion-exchange resin*, and shake for 30 min.

Chromatographic system

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

Mode: LC

Detector: UV 229 nm

Column: 4.6-mm × 25-cm; packing L1

Flow rate: 1.5 mL/min

Injection volume: 20 µL

System suitability

Sample: *Standard solution*

[NOTE—The relative retention times for naloxone and pentazocine are about 0.3 and 1.0, respectively.]

Suitability requirements

Resolution: NLT 6 between pentazocine and naloxone

Relative standard deviation: NMT 2.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of pentazocine ($C_{19}H_{27}NO$) and naloxone ($C_{19}H_{21}NO_4$) in the portion of Tablets taken:

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

r_U = peak response of pentazocine or naloxone from the *Sample solution*

r_S = peak response of pentazocine or naloxone from the *Standard solution*

C_S = concentration of the appropriate USP Pentazocine RS or USP Naloxone RS in the *Standard solution* (mg/mL)

C_U = nominal concentration of pentazocine or naloxone in the *Sample solution* (mg/mL)

Acceptance criteria: 90.0%–110.0% of the labeled amounts of pentazocine ($C_{19}H_{27}NO$) and naloxone ($C_{19}H_{21}NO_4$)

PERFORMANCE TESTS

• **DISSOLUTION ⟨711⟩**

Medium: Water; 900 mL

Apparatus 2: 50 rpm

Time: 45 min

Detector: UV 279 nm (corrected for absorbance at 305 nm)

Standard solution: Dissolve a suitable amount of USP Pentazocine RS in a minimum volume of 0.1 N hydrochloric acid (about 25 mg/mL), diluting quantitatively and stepwise with water.

Sample solution: Filter portions of the solution under test, suitably diluted with *Medium*, if necessary.

Analysis: Determine the labeled amount of pentazocine ($C_{19}H_{27}NO$) dissolved in the *Sample solution* in comparison with the *Standard solution*.

Tolerances: NLT 75% (*Q*) of the labeled amount of pentazocine ($C_{19}H_{27}NO$) is dissolved.

Change to read:

• **UNIFORMITY OF DOSAGE UNITS ⟨905⟩:** ▲Meet the requirements▲ (CN 1-Aug-2023)

Procedure for content uniformity

Diluent: Methanol, water, and phosphoric acid (500:500:1)

Solution A: Prepare a filtered and degassed mixture by dissolving 675 mg of sodium 1-octanesulfonate and 426 mg of anhydrous dibasic sodium phosphate in 625 mL of water, and mix.

Mobile phase: Add 475 mL of methanol and 10 mL of phosphoric acid to *Solution A*.

Strong anion-exchange resin: Transfer 30 g of strong anion-exchange resin to a 250-mL beaker. Wash the resin with two 200-mL portions of water, decanting the water after each wash. Wash with two 200-mL portions of dilute glacial acetic acid (1 in 20), decanting the first wash, and filter with the aid of suction.

Standard stock solution: 0.2 mg/mL of USP Naloxone RS in *Diluent*

Standard solution: Transfer 100 mg of USP Pentazocine RS to a 50-mL volumetric flask. Dissolve in about 30 mL of *Diluent*. Add 5.0 mL of the *Standard stock solution*, and dilute with *Diluent* to volume.

Sample solution: Transfer 1 Tablet to a 25-mL glass-stoppered cylinder. Add 25.0 mL of *Diluent*. Sonicate for 10 min, and shake intermittently for 15 min. Filter into a glass-stoppered conical flask. Add about 125 mg of *Strong anion-exchange resin*, and shake for 30 min.

Chromatographic system

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

Mode: LC

Detector: UV 229 nm

Column: 4.6-mm × 25-cm; packing L1

Flow rate: 1.5 mL/min

Injection volume: 20 µL

System suitability

Sample: *Standard solution* [NOTE—The relative retention times for naloxone and pentazocine are about 0.3 and 1.0, respectively.]

Suitability requirements

Resolution: NLT 6 between pentazocine and naloxone

Relative standard deviation: NMT 2.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of pentazocine ($C_{19}H_{27}NO$) and naloxone ($C_{19}H_{21}NO_4$) in the portion of Tablets taken:

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

r_U = peak response of pentazocine or naloxone from the *Sample solution*

r_S = peak response of pentazocine or naloxone from the *Standard solution*

C_S = concentration of the appropriate USP Pentazocine RS or USP Naloxone RS in the *Standard solution* (mg/mL)

C_U = nominal concentration of pentazocine or naloxone in the *Sample solution* (mg/mL)

▲▲ (CN 1-Aug-2023)

ADDITIONAL REQUIREMENTS

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

USP Naloxone RS

USP Pentazocine RS

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

Topic/Question	Contact	Expert Committee
PENTAZOCINE AND NALOXONE TABLETS	Documentary Standards Support	SM22020 Small Molecules 2
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM22020 Small Molecules 2

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
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