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Pentazocine and Acetaminophen Tablets

DEFINITION

Pentazocine and Acetaminophen Tablets contain an amount of Pentazocine Hydrochloride equivalent to NLT 90.0% and NMT 110.0% of the labeled amount of pentazocine ($C_{19}H_{27}NO$) and NLT 90.0% and NMT 110.0% of the labeled amount of acetaminophen ($C_8H_9NO_2$).

IDENTIFICATION

• **A. Thin-Layer Chromatographic Identification Test ⟨201⟩**

Diluent: Chloroform and methanol (1:1)

Standard solution A: 1 mg/mL of USP Pentazocine RS in *Diluent*

Standard solution B: 26 mg/mL of USP Acetaminophen RS in *Diluent*

Sample solution: Transfer a quantity of finely powdered Tablets, nominally equivalent to about 5 mg of pentazocine and 130 mg of acetaminophen, to a suitable flask. Add 5 mL of *Diluent*, shake, and allow the solids to settle. Use the supernatant.

Chromatographic system

Developing solvent system: Ethyl acetate, methanol, and formic acid (90:5:5)

Spray reagent: Dissolve 300 mg of platinic chloride in 100 mL of water and add 100 mL of potassium iodide solution (6 in 100).

Analysis: Evaporate the solvents in cool, circulating air. After developing and examining the spots, spray the plate with *Spray reagent*.

Acceptance criteria: The R_F values, size, and intensity of color of the two principal spots of the *Sample solution* correspond to those of *Standard solution A* and *Standard solution B*.

ASSAY

• **Pentazocine**

Mobile phase: Chloroform, methanol, and isopropylamine (960:40:2)

Diluent: Methanol and 0.035 N sulfuric acid (1:1)

Standard stock solution: 0.5 mg/mL of USP Pentazocine RS in *Diluent*

Standard solution: Transfer 10.0 mL of the *Standard stock solution* to a 125-mL separator. Add 30 mL of water and 5 mL of sodium carbonate solution (1:10). Extract with 60 mL of chloroform and pass the chloroform layer through filter paper, collecting the filtrate in a 100-mL volumetric flask. Dilute with chloroform to volume and mix.

Sample solution: Transfer an amount nominally equivalent to 25 mg of pentazocine, from NLT 20 finely powdered Tablets, to a 50-mL glass-stoppered cylinder. Add 50.0 mL of *Diluent* and shake intermittently for 15 min. Sonicate for about 2 min, allow the solids to settle, and transfer 10.0 mL of the supernatant to a 125-mL separator. [Note—Save the remainder of the supernatant for use in the *Assay* for *Acetaminophen*. Minimize the waiting period before this test is performed to prevent significant hydrolysis of acetaminophen to *p*-aminophenol.]

Add 30 mL of water and 5 mL of sodium carbonate solution (1:10) to the separator and mix. Extract with 60 mL of chloroform and pass the chloroform layer through filter paper, collecting the filtrate in a 100-mL volumetric flask. Dilute with chloroform to volume and mix.

Chromatographic system

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

Mode: LC

Detector: UV 280 nm

Column: 4.6-mm × 25-cm; 10-µm packing L3

Flow rate: 1.2 mL/min

Injection volume: 20 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Tailing factor: NMT 3.0

Relative standard deviation: NMT 2.0%

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Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of pentazocine ($C_{19}H_{27}NO$) 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 from the *Sample solution*

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

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

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

Acceptance criteria: 90.0%–110.0%

- **Acetaminophen**

[Note—Minimize the time between the addition of the *Diluent* and the injection of the *Sample solution* to prevent significant hydrolysis of acetaminophen to *p*-aminophenol.]

Mobile phase: Chloroform, methanol, and isopropylamine (960:40:2)

Diluent: Methanol and 0.035 N sulfuric acid (1:1)

Standard stock solution: 13 mg/mL of USP Acetaminophen RS in *Diluent*

Standard solution: Dilute 2.0 mL of the *Standard stock solution* with ethyl acetate to 200 mL.

Sample solution: Dilute 2.0 mL of the supernatant reserved from the *Assay* for *Pentazocine* immediately with ethyl acetate to volume in a 200-mL volumetric flask to minimize hydrolysis of acetaminophen to *p*-aminophenol and mix.

Chromatographic system

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

Mode: LC

Detector: UV 254 nm

Column: 4.6-mm × 25-cm; 10-µm packing L3

Flow rate: 1.4 mL/min

Injection volume: 10 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Tailing factor: NMT 3.0

Relative standard deviation: NMT 2.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of acetaminophen ($C_8H_9NO_2$) in the portion of Tablets taken:

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

r_U = peak response of acetaminophen from the *Sample solution*

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

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

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

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

Change to read:

- **Uniformity of Dosage Units ⟨905⟩:** ▲Meet the requirements▲ (CN 1-Aug-2023)

Procedure for content uniformity

Diluent: Acetonitrile and 0.035 N sulfuric acid (6:4)

Mobile phase: Tetrahydrofuran, phosphoric acid, and 0.005 M monobasic sodium phosphate (50:1:950)

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Pentazocine standard stock solution: 0.25 mg/mL of USP Pentazocine RS in *Diluent*

System suitability stock solution: 0.325 mg/mL of USP Acetaminophen RS in *Diluent*

System suitability solution: Transfer 1.0 mL of the *System suitability stock solution* to a 100-mL volumetric flask. Add 5.0 mL of the *Pentazocine standard stock solution*, dilute with *Mobile phase* to volume, and mix.

Standard solution: Transfer a quantity of USP Acetaminophen RS to a suitable volumetric flask. Add a sufficient volume of *Pentazocine standard stock solution* and mix to dissolve the acetaminophen. Dilute with *Mobile phase* to volume. Mix to obtain known concentrations of 0.0125 and 0.325 mg/mL of pentazocine and acetaminophen, respectively.

Sample stock solution: Transfer 1 Tablet to a 100-mL volumetric flask, add 50 mL of *Diluent*, and sonicate for 30 min. Dilute with *Diluent* to volume, and mix. Pass a portion of this solution through a paper filter, covering the funnel with a watch glass and discarding the first few mL of the filtrate.

Sample solution: Dilute 5.0 mL of the *Sample stock solution* with *Mobile phase* to 100 mL and pass this solution through a membrane filter of 0.5-µm or finer pore size.

Chromatographic system

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

Mode: LC

Detector: UV 220 nm

Column: 9.4-mm × 10-cm; 5-µm packing L1

Flow rate: 1.5 mL/min

Injection volume: 10 µL

System suitability

Sample: *System suitability solution*

[NOTE—The relative retention times for acetaminophen and pentazocine are 0.2 and 1.0, respectively.]

Suitability requirements

Resolution: NLT 7 between pentazocine and acetaminophen

Relative standard deviation: NMT 2.0% for the pentazocine and acetaminophen peaks

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of pentazocine ($C_{19}H_{27}NO$) and acetaminophen ($C_8H_9NO_2$) 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 acetaminophen from the *Sample solution*

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

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

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

▲▲ (CN 1-Aug-2023)

IMPURITIES

• **4-AMINOPHENOL IN ACETAMINOPHEN-CONTAINING DRUG PRODUCTS ⟨227⟩:** Meet the requirements

ADDITIONAL REQUIREMENTS

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

• **USP REFERENCE STANDARDS ⟨11⟩**

USP Acetaminophen RS

USP Pentazocine RS

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Topic/Question	Contact	Expert Committee
PENTAZOCINE AND ACETAMINOPHEN TABLETS	Documentary Standards Support	SM22020 Small Molecules 2
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