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Pimozide Tablets

DEFINITION
Pimozide Tablets contain NLT 90.0% and NMT 110.0% of the labeled amount of pimozide ($C_{28}H_{29}F_2N_3O$).

IDENTIFICATION

Change to read:

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

Sample: Grind an appropriate number of Tablets to prepare a 1-mg/mL solution of pimozide in dichloromethane. Shake the solution for 5 min, and pass through a suitable filter. Evaporate the filtrate to dryness under reduced pressure. Use the dried residue.

Acceptance criteria: Meet the requirements

• **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**

Protect all pimozide solutions from light.

Solution A: 2.5 g/L of ammonium acetate and 8.5 g/L of tetrabutylammonium hydrogen sulfate in water

Solution B: Acetonitrile

Mobile phase: See *Table 1*.

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	80	20
10	70	30
15	70	30
16	80	20
30	80	20

System suitability solution: 0.04 mg/mL of USP Pimozide RS and 0.02 mg/mL of USP Mebendazole RS in methanol

Standard solution: 0.4 mg/mL of USP Pimozide RS in methanol

Sample solution: Nominally 0.4 mg/mL of pimozide prepared as follows. Transfer a suitable number of powdered Tablets (NLT 20) to a suitable volumetric flask. Add about 70% of the flask volume of methanol, and mechanically shake for 30 min. Dilute with methanol to volume, and mix well with the aid of sonication for 10 min. Centrifuge, and pass a portion of the supernatant through a suitable filter of 0.45-µm pore size.

Chromatographic system

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

Mode: LC

Detector: UV 280 nm

Column: 4.6-mm × 10-cm; 3-µm packing L1

Flow rate: 2 mL/min

Injection volume: 10 µL

System suitability

Samples: *System suitability solution* and *Standard solution*

[NOTE—The relative retention times for mebendazole and pimozide are 0.88 and 1.0, respectively.]

Suitability requirements

Resolution: NLT 5.0 between the pimozide and mebendazole peaks, *System suitability solution*

Relative standard deviation: NMT 2.0% for pimozide, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of pimozide ($C_{28}H_{29}F_2N_3O$) in the portion of Tablets taken:

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

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

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

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

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

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

• **DISSOLUTION ⟨711⟩**

Medium: 0.01 N hydrochloric acid; 900 mL

Apparatus 2: 50 rpm

Time: 30 min

Buffer: 5 g/L of ammonium acetate in water. Adjust with glacial acetic acid to a pH of 4.5.

Mobile phase: Acetonitrile and *Buffer* (40:60)

Standard solution: (*L*/900) mg/mL of USP Pimozide RS in *Mobile phase*, where *L* is the label claim of pimozide in mg/Tablet

Sample solution: Pass a portion of the solution under test through a suitable filter of 0.45-µm pore size.

Chromatographic system

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

Mode: LC

Detector: UV 280 nm

Column: 3.0-mm × 25-cm; 5-µm packing L1

Flow rate: 0.8 mL/min

Injection volume: 100 µL

Run time: 2.5 times the retention time of pimozide

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 the labeled amount of pimozide ($C_{28}H_{29}F_2N_3O$) dissolved:

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

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

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

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

V = volume of *Medium*, 900 mL

L = label claim (mg/Tablet)

Tolerances: NLT 80% (*Q*) of the labeled amount of pimozide ($C_{28}H_{29}F_2N_3O$) is dissolved.

• **Uniformity of Dosage Units** ⟨905⟩, *Content Uniformity*: Meet the requirements

IMPURITIES

• **Organic Impurities**

Protect all pimozide solutions from light.

Solution A, Solution B, Mobile phase, System suitability solution, and **Chromatographic system:** Proceed as directed in the *Assay*.

Standard solution: 0.01 mg/mL of USP Pimozide RS in methanol

Sample solution: Nominally 2 mg/mL of pimozide prepared as follows. Transfer a suitable number of powdered Tablets (NLT 20) to a suitable volumetric flask. Add about 70% of the flask volume of methanol, and mechanically shake for 30 min. Dilute with methanol to volume, and mix well with the aid of sonication for 10 min. Centrifuge, and pass a portion of the supernatant through a suitable filter of 0.45-µm pore size.

System suitability

Samples: *System suitability solution* and *Standard solution*

[Note—The relative retention times for mebendazole and pimozide are 0.88 and 1.0, respectively.]

Suitability requirements

Resolution: NLT 5.0 between pimozide and mebendazole, *System suitability solution*

Relative standard deviation: NMT 5.0%, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of each individual unspecified degradation product in the portion of Tablets taken:

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

r_U = peak response of any individual unspecified degradation product from the *Sample solution*

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

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

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

Acceptance criteria

Any individual degradation product: NMT 1.0%

Total degradation products: NMT 2.0%

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight, light-resistant containers. Store at controlled room temperature.

• **USP Reference Standards** ⟨11⟩

USP Mebendazole RS

USP Pimozide RS

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

Topic/Question	Contact	Expert Committee
PIMOZIDE TABLETS	Documentary Standards Support	SM42020 Small Molecules 4
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM42020 Small Molecules 4

Chromatographic Database Information: Chromatographic Database

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