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Dicyclomine Hydrochloride Oral Solution

DEFINITION

Dicyclomine Hydrochloride Oral Solution contains NLT 95.0% and NMT 105.0% of the labeled amount of dicyclomine hydrochloride ($C_{19}H_{35}NO_2 \cdot HCl$).

IDENTIFICATION

- **A.**

Sample: Transfer a portion of Oral Solution, equivalent to 100 mg of dicyclomine hydrochloride, to a separator containing 10 mL of water and 1 mL of hydrochloric acid. Extract with two 30-mL portions of ether, and discard the ether. Extract the aqueous acid solution with two 30-mL portions of chloroform, transfer the chloroform extracts to a second separator containing 20 mL of water and 1 mL of sodium hydroxide solution (1 in 10), and shake. Filter the chloroform layer through anhydrous sodium sulfate into a suitable container. Add 3 mL of a freshly prepared 1-in-20 solution of acetyl chloride in anhydrous methanol, prepared by cautiously adding acetyl chloride dropwise to anhydrous methanol with stirring. Evaporate under reduced pressure at room temperature until the residue has been thoroughly dried. Use the residue so obtained to prepare a potassium bromide dispersion.

Standard: Use a similarly prepared potassium bromide dispersion of USP Dicyclomine Hydrochloride RS.

Acceptance criteria: The IR absorption spectrum of the *Sample* exhibits maxima and minima at the same wavelengths as those of the *Standard*.

- **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

Change to read:

- **Procedure**

Buffer: Dissolve 2.72 g of monobasic potassium phosphate in 900 mL of water, adjust with 10% sodium hydroxide to a pH of 7.5 ± 0.1, and dilute with water to 1000 mL.

Mobile phase: Acetonitrile and *Buffer* (70:30)

Diluent: Acetonitrile and *Buffer* (35:65)

Standard solution: 0.1 mg/mL of USP Dicyclomine Hydrochloride RS in *Diluent*. [Note—This solution is stable for ▲at least▲ (USP 1-Dec-2020) 2 days.]

Sample solution: Using a “to contain” pipet, transfer a measured volume of Oral Solution, equivalent to 10.0 mg of dicyclomine hydrochloride, to a 100-mL volumetric flask. Rinse the pipet with several small portions of *Diluent*, and add the rinsings to the volumetric flask. Dilute with *Diluent* to volume.

Chromatographic system

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

Mode: LC

Detector: UV 215 nm

Column: 4.6-mm × 15-cm; 3.5-µm packing L7

Flow rate: ▲1▲ (USP 1-Dec-2020) mL/min

Injection volume: 50 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Tailing factor: NMT 1.5

Relative standard deviation: NMT 1.5%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of dicyclomine hydrochloride ($C_{19}H_{35}NO_2 \cdot HCl$) in the portion of Oral Solution taken:

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

r_U = peak area of dicyclomine from the *Sample solution*

r_S = peak area of dicyclomine from the *Standard solution*

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

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

Acceptance criteria: 95.0%–105.0%

IMPURITIES

Add the following:

▲• **Limit of Dicyclomine Related Compound A**

Buffer: Dissolve 2.72 g of monobasic potassium phosphate in 900 mL of water, adjust with phosphoric acid to a pH of 3.5, and dilute with water to 1000 mL.

Solution A: Acetonitrile and *Buffer* (55:45)

Solution B: Acetonitrile and *Buffer* (80:20)

Mobile phase: See *Table 1*.

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	100	0
20	100	0
20.1	0	100
40	0	100
40.1	100	0
50	100	0

Diluent: Acetonitrile and water (35:65)

Standard stock solution: 0.1 mg/mL of USP Dicyclomine Related Compound A RS in *Diluent*. Sonication may be used.

Standard solution: 2.0 µg/mL of USP Dicyclomine Related Compound A RS in *Diluent* from *Standard stock solution*

Sensitivity solution: 1.0 µg/mL of USP Dicyclomine Related Compound A RS in *Diluent* from *Standard solution*

Sample solution: Nominally 1.0 mg/mL of dicyclomine hydrochloride in *Diluent* prepared as follows. Transfer a measured volume of Oral Solution, equivalent to 20 mg of dicyclomine hydrochloride, to a 20-mL volumetric flask. Rinse the pipet with several small portions of *Diluent*, and add the rinsings to the volumetric flask. Dilute with *Diluent* to volume.

Chromatographic system

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

Mode: LC

Detector: UV 215 nm

Column: 4.6-mm × 15-cm; 3.5-µm packing L7

Flow rate: 1 mL/min

Injection volume: 100 µL

System suitability

Samples: *Standard solution* and *Sensitivity solution*

Suitability requirements

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

Signal-to-noise ratio: NLT 10, *Sensitivity solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of dicyclomine related compound A in the portion of Oral Solution taken:

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

r_U = peak response of dicyclomine related compound A from the *Sample solution*

r_S = peak response of dicyclomine related compound A from the *Standard solution*

C_S = concentration of USP Dicyclomine Related Compound A RS in the *Standard solution* (mg/mL)

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

Acceptance criteria: NMT 0.2%▲ (USP 1-Dec-2020)

ADDITIONAL REQUIREMENTS

Change to read:

• **Packaging and Storage:** Preserve in well-closed containers. ▲Store at controlled room temperature.▲ (USP 1-Dec-2020)

Change to read:

• **USP Reference Standards** ⟨11⟩

USP Dicyclomine Hydrochloride RS

▲ USP Dicyclomine Related Compound A RS

[1,1'-Bi(cyclohexane)]-1-carboxylic acid.

$C_{13}H_{22}O_2$ 210.32▲ (USP 1-Dec-2020)

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

Topic/Question	Contact	Expert Committee
DICYCLOMINE HYDROCHLORIDE ORAL SOLUTION	Documentary Standards Support	SM32020 Small Molecules 3

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