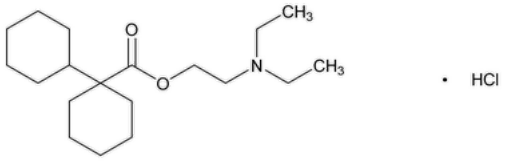
Status: Currently Official on 14-Feb-2025
Official Date: Official as of 01-Feb-2024
Document Type: USP Monographs
DocId: GUID-EA1F263E-7DB5-4B10-A3AE-B02CD628D7ED_4_en-US
DOI: https://doi.org/10.31003/USPNF_M25120_04_01
DOI Ref: ag8op

Dicyclomine Hydrochloride



$C_{19}H_{35}NO_2 \cdot HCl$ 345.95

[Bicyclohexyl]-1-carboxylic acid, 2-(diethylamino)ethyl ester, hydrochloride;

2-(Diethylamino)ethyl [bicyclohexyl]-1-carboxylate hydrochloride CAS RN®: 67-92-5.

DEFINITION

Dicyclomine Hydrochloride contains NLT 98.0% and NMT 102.0% of dicyclomine hydrochloride ($C_{19}H_{35}NO_2 \cdot HCl$), calculated on the dried basis.

IDENTIFICATION

• **A. Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*:** 197K or 197A

Change to read:

• **B. Identification Tests—General ⟨191⟩, *Chemical Identification Tests, Chloride*:** Meets the requirements ▲of the test for amine hydrochlorides▲

(ERR 1-Feb-2024)

• **C.** 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**

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

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

Diluent: Acetonitrile and water (70:30)

Standard solution: 0.4 mg/mL of USP Dicyclomine Hydrochloride RS in *Diluent*. [Note—This solution is stable for at least 2 days.]

Sample solution: 0.4 mg/mL of Dicyclomine Hydrochloride in *Diluent*

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: 50 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Tailing factor: NMT 1.5

Relative standard deviation: NMT 0.73%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of dicyclomine hydrochloride ($C_{19}H_{35}NO_2 \cdot HCl$) in the portion of Dicyclomine Hydrochloride taken:

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

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

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

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

C_U = concentration of Dicyclomine Hydrochloride in the *Sample solution* (mg/mL)

Acceptance criteria: 98.0%–102.0% on the dried basis

IMPURITIES

• **Organic Impurities**

Buffer, Mobile phase, Diluent, Sample solution, and **Chromatographic system:** Proceed as directed in the *Assay*.

Standard stock solution: Use the *Standard solution* from the *Assay*.

Standard solution: 0.4 µg/mL of USP Dicyclomine Hydrochloride RS in *Diluent* from *Standard stock solution*

Sensitivity solution: 0.2 µg/mL of USP Dicyclomine Hydrochloride RS in *Diluent* from *Standard solution*

System suitability

Samples: *Standard stock solution*, *Standard solution*, and *Sensitivity solution*

[Note—USP Dicyclomine Hydrochloride RS contains dicyclomine-1'-ene as a minor component.]

Suitability requirements

Resolution: NLT 2.0 between dicyclomine and dicyclomine-1'-ene, *Standard stock solution*

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 each individual impurity in the portion of Dicyclomine Hydrochloride taken:

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

r_U = peak response of each impurity from the *Sample solution*

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

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

C_U = concentration of Dicyclomine Hydrochloride in the *Sample solution* (mg/mL)

F = relative response factor (see *Table 1*)

Acceptance criteria: See *Table 1*. The reporting threshold is 0.05%.

Table 1

Name	Relative Retention Time	Relative Response Factor	Acceptance Criteria, NMT (%)
Dicyclomine-1'-ene[a]	0.8	2.9	0.5
Dicyclomine	1.0	—	—
Any individual unspecified impurity	—	1.0	0.10

[a] 2-(Diethylamino)ethyl [1,1'-bi(cyclohexan)]-1'-ene-1-carboxylate.

• **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.

Mobile phase: Acetonitrile and *Buffer* (55:45)

Diluent: Acetonitrile and water (70:30)

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

Standard solution: 3.0 µg/mL of USP Dicyclomine Related Compound A RS in *Diluent* from *Standard stock solution*

Sensitivity solution: 1.5 µg/mL of USP Dicyclomine Related Compound A RS in *Diluent* from *Standard solution*

Sample solution: 2.0 mg/mL of Dicyclomine Hydrochloride in *Diluent*. Sonication may be used.

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 Dicyclomine Hydrochloride 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 = concentration of Dicyclomine Hydrochloride in the *Sample solution* (mg/mL)

Acceptance criteria

Dicyclomine related compound A: NMT 0.20%

Total impurities: The sum of all the impurities found in the *Limit of Dicyclomine Related Compound A* and *Organic Impurities* tests is NMT 0.7%.

SPECIFIC TESTS

- **pH** ⟨791⟩

Sample solution: 10 mg/mL

Acceptance criteria: 5.0–5.5

- **Loss on Drying** ⟨731⟩

Analysis: Dry at 105° for 4 h.

Acceptance criteria: NMT 1.0%

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in well-closed containers.
- **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

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

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

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. PF 43(6)

Current DocID: GUID-EA1F263E-7DB5-4B10-A3AE-B02CD628D7ED_4_en-US

DOI: https://doi.org/10.31003/USPNF_M25120_04_01

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