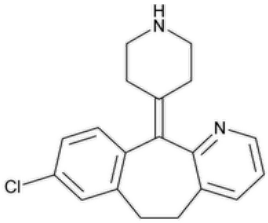
Status: Currently Official on 14-Feb-2025
Official Date: Official as of 01-May-2020
Document Type: USP Monographs
DocId: GUID-94DBE2C4-D40E-4F70-AB75-743A05C0E4F9_2_en-US
DOI: https://doi.org/10.31003/USPNF_M1372_02_01
DOI Ref: 4k4n6

Desloratadine



$C_{19}H_{19}ClN_2$ 310.82

Benzo[5,6]cyclohepta[1,2-*b*]pyridine, 8-chloro-6,11-dihydro-11-(4-piperidinylidene)-, 5*H*-;

8-Chloro-6,11-dihydro-11-(piperidin-4-ylidene)-5*H*-benzo[5,6]cyclohepta[1,2-*b*]pyridine CAS RN®: 100643-71-8; UNII: FVF865388R.

DEFINITION

Desloratadine contains NLT 98.0% and NMT 102.0% of desloratadine ($C_{19}H_{19}ClN_2$), calculated on the anhydrous and solvent-free basis.

IDENTIFICATION

Change to read:

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

• **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**

Buffer: Dissolve 0.865 g of sodium dodecyl sulfate in water, add 0.5 mL of trifluoroacetic acid, and dilute with water to 1 L.

Mobile phase: Acetonitrile and *Buffer* (43:57)

System suitability solution: 0.08 mg/mL of USP Desloratadine RS and 0.2 µg/mL of USP Desloratadine Related Compound B RS in *Mobile phase*

Standard solution: 0.08 mg/mL of USP Desloratadine RS in *Mobile phase*

Sample solution: 0.08 mg/mL of Desloratadine in *Mobile phase*

Chromatographic system

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

Mode: LC

Detector: UV 280 nm

Column: 4.6-mm × 25-cm; 4-µm packing L1

Column temperature 35°

Flow rate: 1 mL/min

Injection volume: 100 µL

Run time: NLT 2 times the retention time of desloratadine

System suitability

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

Suitability requirements

Resolution: NLT 2.0 between desloratadine related compound B and desloratadine, *System suitability solution*

Tailing factor: NMT 1.5, *Standard solution*

Relative standard deviation: NMT 0.73%, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of desloratadine ($C_{19}H_{19}ClN_2$) in the portion of Desloratadine taken:

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

r_U = peak response from the *Sample solution*

r_S = peak response from the *Standard solution*

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

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

Acceptance criteria: 98.0%–102.0% on the anhydrous and solvent-free basis

IMPURITIES

• **Residue on Ignition ⟨281⟩:** NMT 0.2%

• **Organic Impurities, Procedure 1**

Use *Organic Impurities, Procedure 1*, when the impurity profile includes desloratadine related compound B or fluorodesloratadine.

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

Standard solution: 0.08 µg/mL of USP Desloratadine RS in *Mobile phase*

System suitability

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

Suitability requirements

Resolution: NLT 2.0 between desloratadine related compound B and desloratadine, *System suitability solution*

Tailing factor: NMT 1.5, *Standard solution*

Relative standard deviation: NMT 10.0%, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of each impurity in the portion of Desloratadine 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 desloratadine from the *Standard solution*

C_S = concentration of USP Desloratadine RS in the *Standard solution* (µg/mL)

C_U = concentration of Desloratadine in the *Sample solution* (µg/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 (%)
Fluorodesloratadine[a]	0.8	0.6	0.2
Desloratadine related compound B	0.9	0.6	0.3
Desloratadine	1.0	—	—
Any other individual unspecified impurity	—	1.00	0.10
Total impurities	—	—	0.4

[a] 8-Chloro-11-fluoro-11-(piperidin-4-yl)-6,11-dihydro-5*H*-benzo[5,6]cyclohepta[1,2-*b*]pyridine.

• **Organic Impurities, Procedure 2**

Use *Organic Impurities, Procedure 2*, when the impurity profile includes dechloro desloratadine, desloratadine related compound A, or dehydrodesloratadine.

Buffer: 1.36 g/L of monobasic potassium phosphate in water. Add 10 mL of triethylamine per L of the solution, and adjust with dilute phosphoric acid (1 in 10) to a pH of 2.0.

Solution A: Acetonitrile, methanol, and *Buffer* (10:10:80)

Solution B: Acetonitrile, tetrahydrofuran, and *Buffer* (70:5:30)

Mobile phase: See *Table 2*.

Table 2

Time (min)	Solution A (%)	Solution B (%)
0	100	0
10	100	0
15	90	10
20	70	30
25	60	40
30	50	50
38	50	50
40	100	0
45	100	0

System suitability solution: 0.15 µg/mL of USP Desloratadine Related Compound A RS and 100 µg/mL of USP Desloratadine RS in *Solution A*

Standard solution: 0.5 µg/mL of USP Desloratadine RS in *Solution A*

Sample solution: 500 µg/mL of Desloratadine in *Solution A*

Chromatographic system

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

Mode: LC

Detector: UV 280 nm

Column: 4.6-mm × 25-cm; 5-µm packing L7

Flow rate: 1 mL/min

Injection volume: 60 µL

System suitability

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

Suitability requirements

Resolution: NLT 3.0 between desloratadine and desloratadine related compound A, *System suitability solution*

Tailing factor: NMT 3.0 for desloratadine, *System suitability solution*

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

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of each impurity in the portion of Desloratadine 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 desloratadine from the *Standard solution*

C_S = concentration of USP Desloratadine RS in the *Standard solution* (µg/mL)

C_U = concentration of Desloratadine in the *Sample solution* (µg/mL)

F = relative response factor (see *Table 3*)

Acceptance criteria: See *Table 3*.

Table 3

Name	Relative Retention Time	Relative Response Factor	Acceptance Criteria, NMT (%)
Dechloro desloratadine[a]	0.38	0.90	0.15
Desloratadine	1.0	—	—
Desloratadine related compound A	1.30	0.86	0.15
Dehydro desloratadine[b]	1.59	1.00	0.15
Loratadine[c]	2.25	0.79	0.20
Any other individual unspecified impurity	—	1.00	0.10
Total impurities	—	—	0.40

[a] 6,11-Dihydro-11-(piperidin-4-ylidene)-5*H*-benzo[5,6]cyclohepta[1,2-*b*]pyridine.

[b] 8-Chloro-11-(piperidin-4-ylidene)benzo[5,6]cyclohepta[1,2-*b*]pyridine.

[c] 8-Chloro-6,11-dihydro-11-(1-ethoxycarbonylpiperidin-4-ylidene)-5*H*-benzo[5,6]cyclohepta[1,2-*b*]pyridine.

SPECIFIC TESTS

• **Water Determination, *Method Ic*⟨921⟩:** NMT 0.5%

ADDITIONAL REQUIREMENTS

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

• **Labeling:** If a test for *Organic Impurities* other than *Procedure 1* is used, then the labeling states with which *Organic Impurities* test the article complies.

• **USP Reference Standards** ⟨11⟩

USP Desloratadine RS

USP Desloratadine Related Compound A RS

8-Bromo-6,11-dihydro-11-(piperidin-4-ylidene)-5*H*-benzo[5,6]cyclohepta[1,2-*b*]pyridine.

$C_{19}H_{19}BrN_2$ 355.27

USP Desloratadine Related Compound B RS

8-Chloro-11-(1,2,3,6-tetrahydropyridin-4-yl)-6,11-dihydro-5*H*-benzo[5,6]cyclohepta[1,2-*b*]pyridine hydrochloride.

$C_{19}H_{20}Cl_2N_2$ 347.28

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

Topic/Question	Contact	Expert Committee
DESLORATADINE	Documentary Standards Support	SM52020 Small Molecules 5

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. PF 41(2)
Current DocID: GUID-94DBE2C4-D40E-4F70-AB75-743A05C0E4F9_2_en-US
DOI: https://doi.org/10.31003/USPNF_M1372_02_01
DOI ref: 4k4n6