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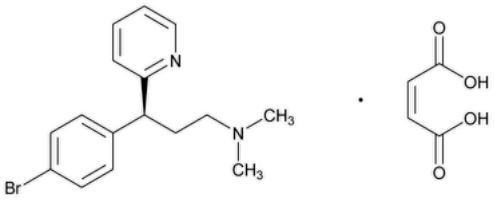
Dexbrompheniramine Maleate



$C_{16}H_{19}BrN_2 \cdot C_4H_4O_4$ 435.32

2-Pyridinepropanamine, γ-(4-bromophenyl)-*N*,*N*-dimethyl-, (*S*)-, (*Z*)-2-butenedioate (1:1);
(+)-2-[*p*-Bromo-α-[2-(dimethylamino)ethyl]benzyl]pyridine maleate (1:1) CAS RN®: 2391-03-9.

DEFINITION

Dexbrompheniramine Maleate contains NLT 98.0% and NMT 102.0% of dexbrompheniramine maleate ($C_{16}H_{19}BrN_2 \cdot C_4H_4O_4$), calculated on the dried basis.

IDENTIFICATION

Change to read:

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

• **B.** The retention times of the maleic acid and dexbrompheniramine peaks of the *Sample solution* correspond to those of the *Standard solution*, as obtained in the *Assay*.

ASSAY

• **Procedure**

Solution A: 5.44 g/L of monobasic potassium phosphate. Adjust with phosphoric acid to a pH of 3.0 ± 0.1.

Solution B: Acetonitrile

Diluent: Acetonitrile and *Solution A* (5:95)

System suitability stock solution: 0.02 mg/mL each of USP Pheniramine Maleate RS, USP Chlorpheniramine Maleate RS, and USP Chlorpheniramine Related Compound B RS in *Diluent*. Sonicate for 1 min.

System suitability solution: 0.5 mg/mL of USP Dexbrompheniramine Maleate RS and 2 µg/mL each of USP Pheniramine Maleate RS, USP Chlorpheniramine Maleate RS, and USP Chlorpheniramine Related Compound B RS in *Diluent*, prepared as follows. Transfer 5.0 mg of USP Dexbrompheniramine Maleate RS to a 10-mL volumetric flask, add 5.0 mL of *Diluent* and 1.0 mL of the *System suitability stock solution*, and dilute with *Diluent* to volume.

Standard solution: 0.5 mg/mL of USP Dexbrompheniramine Maleate RS in *Diluent*. Sonicate for 1 min.

Sample solution: 0.5 mg/mL of Dexbrompheniramine Maleate in *Diluent*. Sonicate for 1 min.

Mobile phase: See *Table 1*.

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	95	5
1	95	5
20	70	30
30	70	30
31	95	5
40	95	5

Chromatographic system

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

Mode: LC

Detector: UV 225 nm

Column: 4.6-mm × 25-cm; 5-µm packing L1

Column temperature: 30°

Flow rate: 1 mL/min

Injection volume: 10 µL

System suitability

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

Suitability requirements

[NOTE—The relative retention times for maleic acid and dexbrompheniramine are 0.18 and 1.0, respectively.]

Resolution: NLT 1.5 between chlorpheniramine and dexbrompheniramine; NLT 2.0 between chlorpheniramine related compound B and pheniramine, *System suitability solution*

Tailing factor: NMT 2.0, *Standard solution*

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

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of dexbrompheniramine maleate ($C_{16}H_{19}BrN_2 \cdot C_4H_4O_4$) in the portion of Dexbrompheniramine Maleate 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 Dexbrompheniramine Maleate RS in the *Standard solution* (mg/mL)

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

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

IMPURITIES

• **RESIDUE ON IGNITION ⟨281⟩:** NMT 0.2%

• **ORGANIC IMPURITIES**

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

Standard solution: 2.7 µg/mL of USP Dexbrompheniramine Maleate RS in *Diluent*, equivalent to 2.0 µg/mL of dexbrompheniramine. Sonicate for 1 min.

Sensitivity solution: 0.74 µg/mL of USP Pheniramine Maleate RS in *Diluent*

Sample solution: 0.5 mg/mL of Dexbrompheniramine Maleate in *Diluent*. Sonicate for 1 min.

System suitability

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

Suitability requirements

Resolution: NLT 1.5 between chlorpheniramine and dexbrompheniramine; NLT 2.0 between chlorpheniramine related compound B and pheniramine, *System suitability solution*

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

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

Analysis

Sample: *Sample solution*

[NOTE—Exclude the maleic acid peak at the relative retention time 0.18.]

Calculate the percentage of each impurity in the portion of Dexbrompheniramine Maleate taken:

$$\text{Result} = (r_U/r_T) \times 100$$

r_U = peak response of each impurity

r_T = sum of the responses of all the peaks

Acceptance criteria

Total impurities: NMT 2.0%

SPECIFIC TESTS

• **LOSS ON DRYING ⟨731⟩**

Analysis: Dry at 65° for 4 h.

Acceptance criteria: NMT 0.5%

- **Optical Rotation,** *Specific Rotation* ⟨781S⟩

Sample solution: 50 mg/mL, in dimethylformamide

Acceptance criteria: +35.0° to +38.5°

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in tight, light-resistant containers.
- **USP Reference Standards** ⟨11⟩

USP Chlorpheniramine Maleate RS

USP Chlorpheniramine Related Compound B RS

Di(pyridin-2-yl)amine.

$C_{10}H_9N_3$ 171.20

USP Dexbrompheniramine Maleate RS

USP Pheniramine Maleate RS

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

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

Chromatographic Database Information: Chromatographic Database

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