Status: Currently Official on 14-Feb-2025
Official Date: Official Prior to 2013
Document Type: USP Monographs
DocId: GUID-7D38798A-7EB0-40C5-9256-291240578864_1_en-US
DOI: https://doi.org/10.31003/USPNF_M23570_01_01
DOI Ref: 5d9f4

Dexbrompheniramine Maleate and Pseudoephedrine Sulfate Oral Solution

» Dexbrompheniramine Maleate and Pseudoephedrine Sulfate Oral Solution contains not less than 90.0 percent and not more than 110.0 percent of the labeled amounts of dexbrompheniramine maleate ($C_{10}H_{15}BrN_2 \cdot C_4H_4O_4$) and pseudoephedrine sulfate [$(C_{10}H_{15}NO)_2 \cdot H_2SO_4$].

USP Reference standards ⟨11⟩—
USP Dexbrompheniramine Maleate RS
USP Pseudoephedrine Sulfate RS

Identification—

A: The retention time of the major peak for dexbrompheniramine maleate in the chromatogram of the *Assay preparation* corresponds to that in the chromatogram of the *Standard preparation,* as obtained in the *Assay.*

B: The retention time of the major peak for pseudoephedrine sulfate in the chromatogram of the *Assay preparation* corresponds to that in the chromatogram of the *Standard preparation,* as obtained in the *Assay.*

C: A solution of it responds to the test for *Sulfate ⟨191⟩*.

D: Transfer a volume of Oral Solution, equivalent to about 6 mg of dexbrompheniramine maleate, to a separatory funnel, add 0.5 mL of ammonium hydroxide and 5 mL of methylene chloride, shake for 1 minute, and allow the layers to separate. Use the clear, lower layer as the test solution. Prepare a Standard solution in methanol containing 1.2 mg of USP Dexbrompheniramine Maleate RS and a second Standard solution in methanol containing 9 mg of USP Pseudoephedrine Sulfate RS per mL. Separately apply 5 µL of the test solution and each Standard solution to a suitable thin-layer chromatographic plate (see *Chromatography ⟨621⟩*) coated with a 0.25-mm layer of chromatographic silica gel mixture. Allow the spots to dry, and develop the chromatogram in a solvent system consisting of a mixture of ethyl ether, methanol, and ammonium hydroxide (16:3:1) until the solvent front has moved about three-fourths of the length of the plate. Remove the plate from the developing chamber, mark the solvent front, and allow the solvent to evaporate. Locate the spots on the plate by examination under short-wavelength UV light: the R_F values of the two principal spots obtained from the test solution correspond to those obtained from the respective Standard solutions.

Uniformity of dosage units ⟨905⟩—
FOR ORAL SOLUTION PACKAGED IN SINGLE-UNIT CONTAINERS: meets the requirements.

Deliverable volume ⟨698⟩—
FOR ORAL SOLUTION PACKAGED IN MULTIPLE-UNIT CONTAINERS: meets the requirements.

Assay—

Mobile phase—Prepare a mixture of water, acetonitrile, methanol, and tetrahydrofuran (55:32:8:5). Transfer 0.1 mL of phosphoric acid, followed by 0.433 g of sodium lauryl sulfate, to each 100 mL of this mixture, and mix. Adjust with ammonium hydroxide to a pH of 3.50 ± 0.05, filter, and degas. Make adjustments if necessary (see *System Suitability* under *Chromatography ⟨621⟩*). [Note—The pH of the *Mobile phase* is critical, and may cause differences of 1 to 4 minutes in the retention times of the internal standard and dexbrompheniramine.]

Internal standard solution—Dissolve an accurately weighed quantity of naphazoline hydrochloride in *Mobile phase* to obtain a solution containing 0.5 mg per mL.

Dexbrompheniramine standard solution—Dissolve an accurately weighed quantity of USP Dexbrompheniramine Maleate RS in *Mobile phase* to obtain a solution having a known concentration of about 6000*J* µg per mL, *J* being the ratio of the labeled amount, in mg, of dexbrompheniramine maleate to the labeled amount, in mg, of pseudoephedrine sulfate per mL of the Oral Solution.

Standard preparation—Transfer about 30 mg of USP Pseudoephedrine Sulfate RS, accurately weighed, to a 25-mL volumetric flask, add 5.0 mL each of *Dexbrompheniramine standard solution* and *Internal standard solution,* dilute with *Mobile phase* to volume, and mix to obtain a *Standard preparation* having known concentrations of about 1.2*J* mg of USP Dexbrompheniramine Maleate RS per mL and about 1.2 mg of USP Pseudoephedrine Sulfate RS per mL.

Assay preparation—Using a "to contain" pipet, transfer an accurately measured volume of Oral Solution, equivalent to about 30 mg of pseudoephedrine sulfate, to a 25-mL volumetric flask. Rinse the pipet with about 5 mL of *Mobile phase*, collecting the rinsing in the volumetric flask. Add 5.0 mL of *Internal standard solution,* dilute with *Mobile phase* to volume, and mix.

Chromatographic system (see *Chromatography* ⟨621⟩)—The liquid chromatograph is equipped with a 254-nm detector and a 4-mm × 30-cm column that contains packing L11. The flow rate is about 1.5 mL per minute. Chromatograph the *Standard preparation,* and record the peak responses as directed for *Procedure:* the relative retention times are about 1.0 for pseudoephedrine, 1.5 for naphazoline, and 2.5 for dexbrompheniramine; the resolution, *R,* between the pseudoephedrine and naphazoline peaks is not less than 3; the resolution, *R,* between the dexbrompheniramine and naphazoline peaks is not less than 3; and the relative standard deviation for replicate injections is not more than 2.0%.

Procedure—Separately inject equal volumes (about 10 µL) of the *Standard preparation* and the *Assay preparation* into the chromatograph, record the chromatograms, and measure the responses for the major peaks. Calculate the quantities, in mg per mL, of dexbrompheniramine maleate ($C_{10}H_{15}BrN_2 \cdot C_4H_4O_4$) and pseudoephedrine sulfate [$(C_{10}H_{15}NO)_2 \cdot H_2SO_4$] in the portion of Oral Solution taken by the formula:

$$25CV(R_U/R_S)$$

in which C is the concentration, in mg per mL, of the appropriate USP Reference Standard in the *Standard preparation; V* is the volume, in mL, of Oral Solution taken; and R_U and R_S are the ratios of the peak responses of the corresponding analyte to the internal standard obtained from the *Assay preparation* and the *Standard preparation,* respectively.

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

We apologize for the inconvenience. The exact auxiliary information for this Documentary Standard is currently unavailable. Please contact Documentary Standards Support (stdsmonographs@usp.org) for assistance during this time.

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. PF 30(1)

Current DocID: GUID-7D38798A-7EB0-40C5-9256-291240578864_1_en-US
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