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Ibuprofen and Pseudoephedrine Hydrochloride Tablets

» Ibuprofen and Pseudoephedrine Hydrochloride Tablets contain not less than 90.0 percent and not more than 110.0 percent of the labeled amounts of Ibuprofen ($C_{13}H_{18}O_2$) and Pseudoephedrine Hydrochloride ($C_{10}H_{15}NO \cdot HCl$).

Packaging and storage—Preserve in tight containers.

USP Reference standards ⟨11⟩—

USP Ibuprofen RS
USP Pseudoephedrine Hydrochloride RS

Identification—

A: Place a Tablet in a small beaker, crack the Tablet coating, add 10 mL of methanol, and stir by mechanical means for about 10 minutes. Allow to settle, and use the clear supernatant as the *Test solution.* Prepare a Standard solution in methanol containing about 20 mg of USP Ibuprofen RS and 20*J* mg of USP Pseudoephedrine Hydrochloride RS per mL, *J* being the ratio of the labeled amount, in mg, of pseudoephedrine hydrochloride to the labeled amount, in mg, of ibuprofen per Tablet. Separately apply 10 µL each of the *Test solution* and the Standard solution to a thin-layer chromatographic plate (see *Chromatography* ⟨621⟩) covered with a 0.25-mm layer of chromatographic silica gel mixture and activated by heating the plate at 105° for about 30 minutes. Place the plate in a chromatographic chamber equilibrated with a solvent system consisting of a mixture of chloroform, methanol, and glacial acetic acid (80:15:5). Develop the chromatograms until the solvent has moved about 10 cm from the origin. Remove the plate from the chromatographic chamber, place it in a chamber containing iodine vapors for about 10 minutes, and examine the chromatograms: the principal spots obtained from the *Test solution* correspond in R_F value and appearance to those obtained from the Standard solution.

B: The retention times of the pseudoephedrine and ibuprofen peaks, relative to that of the butylparaben internal standard peak in the chromatogram of the *Assay preparation* correspond to those in the chromatogram of the *Standard preparation,* as obtained in the *Assay.*

Dissolution ⟨711⟩—

Medium: pH 7.2 phosphate buffer (see under *Buffers* in the section *Reagents, Indicators, and Solutions*); 900 mL.

Apparatus 2: 50 rpm.

Times: 30 minutes (ibuprofen); 45 minutes (pseudoephedrine hydrochloride).

Procedure for ibuprofen—Determine the amount of ibuprofen ($C_{13}H_{18}O_2$) dissolved from UV absorbances at the wavelength of maximum absorbance at about 224 nm of filtered portions of the solution under test, suitably diluted with *Medium*, if necessary, in comparison with a Standard solution having a known concentration of USP Ibuprofen RS in the same medium.

Procedure for pseudoephedrine hydrochloride—

MOBILE PHASE—Prepare a solution of monobasic potassium phosphate in water containing 500 mg per 1000 mL. Filter through a filter having a porosity of 0.5 µm or finer. Prepare a mixture of this solution and acetonitrile (500:500), and adjust with phosphoric acid to a pH of 3.3 ± 0.1. Make any necessary adjustments (see *System Suitability* under *Chromatography* ⟨621⟩). Increasing the concentration of monobasic potassium phosphate or increasing the pH increases the retention time of pseudoephedrine.

STANDARD PREPARATION—Prepare a solution of USP Pseudoephedrine Hydrochloride RS in *Dissolution Medium* having a known concentration of about *P*/900 mg per mL, *P* being the labeled quantity, in mg, of pseudoephedrine hydrochloride per Tablet.

CHROMATOGRAPHIC SYSTEM (see *Chromatography* ⟨621⟩)—The liquid chromatograph is equipped with a 215-nm detector, a guard column containing packing L10, and a 4.6-mm × 25-cm column that contains packing L10. The flow rate is about 1.5 mL per minute. Chromatograph the *Standard preparation*, and record the peak responses as directed for *Procedure:* the tailing factor for the pseudoephedrine peak is not more than 2.0, and the relative standard deviation for replicate injections is not more than 2.0%.

PROCEDURE—Pass a portion of the solution under test through a filter having a porosity of 0.5 µm or finer. Separately inject equal volumes (about 10 µL) of the filtrate and the *Standard preparation* into the chromatograph, record the chromatograms, and measure the areas for the pseudoephedrine peaks. Calculate the quantity, in mg, of pseudoephedrine hydrochloride ($C_{10}H_{15}NO \cdot HCl$) dissolved by the formula:

$$900C(r_U/r_S)$$

in which *C* is the concentration, in mg per mL, of USP Pseudoephedrine Hydrochloride RS in the *Standard preparation;* and r_U and r_S are pseudoephedrine peak responses obtained from the solution under test and the *Standard preparation*, respectively.

Tolerances—Not less than 75% *(Q)* of the labeled amounts of ibuprofen ($C_{13}H_{18}O_2$) and pseudoephedrine hydrochloride ($C_{10}H_{15}NO \cdot HCl$) are dissolved in 30 minutes and in 45 minutes, respectively.

Uniformity of dosage units ⟨905⟩—

Procedure for content uniformity—Proceed as directed in the *Assay*, preparing the *Assay preparation* as follows. Transfer 1 Tablet to a glass-stoppered conical flask, add 10.0 mL of *Internal standard solution*, and stir with a magnetic stirrer until the Tablet disintegrates. Add 10.0 mL of acetonitrile, stir for about 15 minutes, and filter.

Assay—

Mobile phase—Dissolve 2.5 g of docusate sodium in a mixture of water and acetonitrile (590:410). Add 1.0 mL of phosphoric acid, and adjust with ammonium hydroxide to a pH of 3.2 ± 0.05. Make any necessary adjustments (see *System Suitability* under *Chromatography ⟨621⟩*). Decreasing the proportion of docusate sodium increases the resolution between pseudoephedrine and ibuprofen.

Internal standard solution—Prepare a solution of butylparaben in *Mobile phase* containing about 0.15 mg per mL.

Standard preparation—Prepare a solution in *Internal standard solution* having known concentrations of about 20 mg of USP Ibuprofen RS and 20*J* mg of USP Pseudoephedrine Hydrochloride RS per mL, *J* being the ratio of the labeled amount, in mg, of pseudoephedrine hydrochloride to the labeled amount, in mg, of ibuprofen per Tablet. To the resulting solution add an equal volume of acetonitrile, accurately measured, and mix. Pass through a filter having 0.5 µm porosity or finer, and use the filtrate as the *Standard preparation*. This solution contains about 10 mg of USP Ibuprofen RS and 10*J* mg of USP Pseudoephedrine Hydrochloride RS per mL.

Assay preparation—Transfer an accurately counted number of Tablets, equivalent to about 2000 mg of ibuprofen, to a glass-stoppered conical flask, add 100 mL of *Internal standard solution*, and stir with a magnetic stirrer until the Tablets disintegrate. Add 100 mL of acetonitrile, and mix. Filter through a filter of 0.5 µm porosity or finer, and use the filtrate as the *Assay preparation*.

Chromatographic system (see *Chromatography ⟨621⟩*)—The liquid chromatograph is equipped with a 254-nm detector, a guard column that contains packing L1, and a 4.6-mm × 10-cm column that contains 5-µm packing L1. The flow rate is about 2 mL per minute. Chromatograph the *Standard preparation*, and record the peak responses as directed for *Procedure:* the relative retention times are about 0.55 for butylparaben, 0.7 for pseudoephedrine, and 1.0 for ibuprofen; the resolution, *R,* between the butylparaben peak and the pseudoephedrine peak and between the pseudoephedrine peak and the ibuprofen peak is not less than 2.0; the tailing factors for the butylparaben peak, the pseudoephedrine peak, and the ibuprofen peak are not more than 3.0; and the relative standard deviation for replicate injections determined from the peak response ratios is not more than 2.0%.

Procedure—Separately inject equal volumes (about 5 µL) of the *Standard preparation* and the *Assay preparation* into the chromatograph, record the chromatograms, and measure the areas for the major peaks. Calculate the quantities, in mg, of ibuprofen ($C_{13}H_{18}O_2$) and of pseudoephedrine hydrochloride ($C_{10}H_{15}NO \cdot HCl$) in each Tablet taken by the formula:

$$200(C/N)(R_U/R_S)$$

in which *C* is the concentration, in mg per mL, of USP Ibuprofen RS or USP Pseudoephedrine Hydrochloride RS, as appropriate, in the *Standard preparation; N* is the number of Tablets taken; and R_U and R_S are the ratios of the relevant analyte peak response to the butylparaben peak response obtained from the *Assay preparation* and the *Standard preparation*, respectively.

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

Topic/Question	Contact	Expert Committee
IBUPROFEN AND PSEUDOEPHEDRINE HYDROCHLORIDE TABLETS	Documentary Standards Support	SM22020 Small Molecules 2
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM22020 Small Molecules 2

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
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