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Pyrazinamide Tablets

» Pyrazinamide Tablets contain not less than 93.0 percent and not more than 107.0 percent of the labeled amount of pyrazinamide ($C_5H_5N_3O$).

Packaging and storage—Preserve in well-closed containers.

USP Reference standards ⟨11⟩—

USP Pyrazinamide RS

Identification—

A: To a quantity of powdered Tablets, equivalent to about 1 g of pyrazinamide, add about 75 mL of isopropyl alcohol, heat on a steam bath, and filter while hot. Allow to cool, filter the crystals that form, and dry at 105° for 1 hour: the IR absorption spectrum of a mineral oil dispersion of the dried crystals so obtained exhibits maxima only at the same wavelengths as that of a similar preparation of USP Pyrazinamide RS. If a difference appears, dissolve portions of both the dried crystals and the Reference Standard in acetone, evaporate the solutions to dryness, and repeat the test on the residues.

B: The dried crystals obtained in *Identification* test *A* meet the requirements for *Identification* test *B* under *Pyrazinamide*.

C: To 20 mg of the dried crystals obtained in *Identification* test *A* add 5 mL of 5 N sodium hydroxide, and heat gently over an open flame: the odor of ammonia is perceptible.

Dissolution ⟨711⟩—

Medium: water; 900 mL.

Apparatus 2: 50 rpm.

Time: 45 minutes.

Procedure—Determine the amount of $C_5H_5N_3O$ dissolved by employing UV absorption at the wavelength of maximum absorbance at about 268 nm on filtered portions of the solution under test, suitably diluted with *Dissolution Medium*, if necessary, in comparison with a Standard solution having a known concentration of USP Pyrazinamide RS in the same *Medium*.

Tolerances—Not less than 75% *(Q)* of the labeled amount of $C_5H_5N_3O$ is dissolved in 45 minutes.

Uniformity of dosage units ⟨905⟩: meet the requirements.

Assay—

Mobile phase—Prepare pH 8.0 phosphate buffer (see *Buffer Solutions* in the section *Reagents, Indicators, and Solutions*), and adjust with phosphoric acid to a pH of 3.0. Mix 10 mL of acetonitrile with 1 L of this solution, filter, and degas. Make adjustments if necessary (see *System Suitability* under *Chromatography* ⟨621⟩).

Standard preparation—Transfer an accurately weighed quantity of USP Pyrazinamide RS to a suitable volumetric flask, dissolve in water, sonicating to dissolve, dilute with water to volume, and mix to obtain a solution having a known concentration of about 0.1 mg per mL. Transfer 20.0 mL of the solution to a 50-mL volumetric flask, dilute with water to volume, and mix.

System suitability solution—Transfer 1 mL of hydrochloric acid to a 5-mL volumetric flask, dilute with *Standard preparation* to volume, and mix. Keep this solution on a boiling water bath for 5 minutes, and cool.

Assay preparation—Accurately weigh not fewer than 20 Tablets, and grind to a fine powder. Transfer an accurately weighed quantity of the powder, equivalent to about 100 mg of pyrazinamide, to a 500-mL volumetric flask, add 300 mL of water, and sonicate for 10 minutes. Dilute with water to volume, and mix. Filter a portion of this solution, discarding the first few mL of the filtrate. Transfer 20.0 mL of this filtrate to a 100-mL volumetric flask, dilute with water to volume, and mix.

Chromatographic system (see *Chromatography* ⟨621⟩)—The liquid chromatograph is equipped with a 270-nm detector and a 3.9-mm × 15-cm column that contains packing L1. The flow rate is about 1 mL per minute. Chromatograph the *Standard preparation*, and record the peak responses as directed for *Procedure:* the column efficiency is not less than 2500 theoretical plates; and the tailing factor for the pyrazinamide peak is not more than 1.3. Chromatograph the *System suitability solution*, and record the peak responses as directed for *Procedure:* the relative retention times are about 0.45 for pyrazinoic acid and 1.0 for pyrazinamide; and the resolution, *R,* between pyrazinamide and pyrazinoic acid is not less than 6.0.

Procedure—Separately inject equal volumes (about 20 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 quantity, in mg, of pyrazinamide ($C_5H_5N_3O$) in the

portion of Tablets taken by the formula:

$$2.5C(r_U/r_S)$$

in which C is the concentration, in µg, of USP Pyrazinamide RS in the *Standard preparation;* and r_U and r_S are the peak responses 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
PYRAZINAMIDE TABLETS	Documentary Standards Support	SM12020 Small Molecules 1
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM12020 Small Molecules 1

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. Information currently unavailable

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