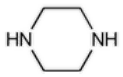
Status: Currently Official on 16-Feb-2025
Official Date: Official as of 01-May-2020
Document Type: USP Monographs
DocId: GUID-7E838D5C-328A-4F98-A8A8-EBACFE436797_4_en-US
DOI: https://doi.org/10.31003/USPNF_M65240_04_01
DOI Ref: ubu2o

Piperazine



$C_4H_{10}N_2$ 86.14

Piperazine.

Piperazine CAS RN®: 110-85-0; UNII: 1RTM4PAL0V.

» Piperazine contains not less than 98.0 percent and not more than 101.0 percent of $C_4H_{10}N_2$, calculated on the anhydrous basis.

Packaging and storage—Preserve in tight containers, protected from light.

USP Reference standards ⟨11⟩—

USP Piperazine RS

Color of solution—Dissolve 10.0 g in water, and dilute with water to 50.0 mL: the solution has no more color than a standard solution prepared by adding 2.0 mL of ferric chloride CS to water and diluting with water to 50.0 mL, when compared in matched color-comparison tubes.

Identification—

Change to read:

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

B: In the test for *Chromatographic purity,* the principal spot in the chromatogram of *Test solution 2,* observed after spraying with the ninhydrin solutions, corresponds in R_F value, color, and size to that in the chromatogram of *Standard solution 1*.

Melting range ⟨741⟩: between 109° and 113°.

Water Determination, *Method I* ⟨921⟩: not more than 2.0%.

Chromatographic purity—

Solvent—Prepare a mixture of 13.5 N ammonium hydroxide and dehydrated alcohol (3:2).

Standard solution 1—Prepare a solution of USP Piperazine RS in *Solvent* containing 10 mg per mL.

Standard solution 2—Prepare a solution of ethylenediamine in *Solvent* containing 0.25 mg per mL.

Standard solution 3—Prepare a solution of triethylenediamine in *Solvent* containing 0.25 mg per mL.

Resolution solution—Prepare a solution in *Solvent* containing 0.25 mg of triethylenediamine and 10 mg of USP Piperazine RS per mL.

Test solution 1—Prepare a solution of Piperazine in *Solvent* containing 100 mg per mL.

Test solution 2—Mix 1 mL of *Test solution 1* and 9 mL of *Solvent*.

Procedure—Apply separate 5-µL portions of *Standard solution 1, Standard solution 2, Standard solution 3, Resolution solution, Test solution 1,* and *Test solution 2* to a suitable thin-layer chromatographic plate (see *Chromatography ⟨621⟩*), coated with a 0.25-mm layer of chromatographic silica gel. Allow the spots to dry, and develop the chromatograms in a solvent system consisting of a freshly prepared mixture of acetone and 13.5 N ammonium hydroxide (80:20) 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 dry the plate at 105°. Spray the plate with a 0.3% (w/v) solution of ninhydrin in a mixture of butyl alcohol and glacial acetic acid (100:3). Spray the plate again with a 0.15% (w/v) solution of ninhydrin in dehydrated alcohol, dry the plate at 105° for 10 minutes, and examine the plate: any secondary spot in the chromatogram obtained from *Test solution 1* is not more intense than the principal spot in the chromatogram obtained from *Standard solution 2* (0.25%). Spray the plate with 0.1 N iodine TS, allow to stand for 10 minutes, and examine the plate: any spot corresponding to triethylenediamine in the chromatogram obtained from *Test solution 1* is not more intense than the principal spot in the chromatogram obtained from *Standard solution 3* (0.25%). In a valid test, the chromatogram obtained from the *Resolution solution* shows a spot due to triethylenediamine clearly separated from the principal spot. Disregard any spot at the origin of any chromatogram.

Assay—Weigh accurately about 150 mg of Piperazine, and dissolve in 75 mL of glacial acetic acid. Titrate potentiometrically with 0.1 N perchloric acid VS, using a silver-glass electrode system. As the endpoint is approached, warm the solution to 60° to 70°, then complete the

titration. Perform a blank determination, and make any necessary correction. Each mL of 0.1 N perchloric acid is equivalent to 4.307 mg of $C_4H_{10}N_2$.

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

Topic/Question	Contact	Expert Committee
PIPERAZINE	Documentary Standards Support	SM32020 Small Molecules 3
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM32020 Small Molecules 3

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. PF 34(1)

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