pH ⟨791⟩: between 5.0 and 6.6, in a solution (1 in 20).

Loss on drying ⟨731⟩—Dry it at 105° for 4 hours: it loses not more than 0.5% of its weight.

Residue on ignition ⟨281⟩: not more than 0.1%.

Chromatographic purity—

Standard solutions—Dissolve USP Xylometazoline Hydrochloride RS in methanol, and mix to obtain *Standard preparation A* having a known concentration of 100 µg per mL. Dilute quantitatively with methanol to obtain *Standard solutions*, designated below by letter, having the following compositions:

Standard solution	Dilution	Concentration (µg RS per mL)	Percentage (%, for comparison with test specimen)
A	(undiluted)	100	0.5
B	(4 in 5)	80	0.4
C	(3 in 5)	60	0.3
D	(2 in 5)	40	0.2
E	(1 in 5)	20	0.1

Test solution—Dissolve an accurately weighed quantity of Xylometazoline Hydrochloride in methanol to obtain a solution containing 20 mg per mL.

Identification solution—Dilute a portion of the *Test solution* quantitatively with methanol to obtain a solution containing 100 µg per mL.

Detection reagent—Prepare (1) a solution of 0.5 g of potassium iodide in 50 mL of water, and (2) a solution of 1.5 g of soluble starch in 50 mL of boiling water. Just prior to use, mix 10 mL of each solution with 3 mL of alcohol.

Procedure—Apply separately 5 µL of the *Test solution*, 5 µL of the *Identification solution*, and 5 µL of each *Standard solution* to a suitable thin-layer chromatographic plate (see *Chromatography* ⟨621⟩) coated with a 0.25-mm layer of chromatographic silica gel. Position the plate in a chromatographic chamber, and develop the chromatograms in a solvent system consisting of a mixture of methanol and ammonium hydroxide (20:1) 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 plate to dry under a current of warm air for at least 30 minutes. Expose the plate to chlorine gas for not more than 5 minutes, and air-dry until the chlorine has dissipated (about 15 minutes). Spray the plate with *Detection reagent*, and immediately compare the intensities of any secondary spots observed in the chromatogram of the *Test solution* with those of the principal spots in the chromatograms of the *Standard solutions:* the sum of the intensities of all secondary spots obtained from the *Test solution* corresponds to not more than 1.0%.

Assay—Dissolve about 500 mg of Xylometazoline Hydrochloride, accurately weighed, in 70 mL of glacial acetic acid, add 10 mL of mercuric acetate TS, and titrate with 0.1 N perchloric acid VS, determining the endpoint potentiometrically (see *Titrimetry* ⟨541⟩), using a calomel-glass electrode system. Perform a blank determination, and make any necessary correction. Each mL of 0.1 N perchloric acid is equivalent to 28.08 mg of $C_{16}H_{24}N_2 \cdot HCl$.

Xylometazoline Hydrochloride Nasal Solution

» Xylometazoline Hydrochloride Nasal Solution is an isotonic solution of Xylometazoline Hydrochloride in Water. It contains not less than 90.0 percent and not more than 110.0 percent of the labeled amount of xylometazoline hydrochloride ($C_{16}H_{24}N_2 \cdot HCl$).

Packaging and storage—Preserve in tight, light-resistant containers.

USP Reference standards ⟨11⟩—
USP Xylometazoline Hydrochloride RS

Identification—

Standard solution—Dissolve an accurately weighed quantity of USP Xylometazoline Hydrochloride RS in water to obtain a solution having a known concentration of about 1 mg per mL, and proceed as directed for *Test solution*.

Test solution—Transfer 10 mL to a suitable separator, add 2 mL of sodium carbonate solution (1 in 10), and extract with 10 mL of chloroform, filtering the extract through anhydrous sodium sulfate. Evaporate the chloroform extract on a steam bath to dryness, and dissolve the residue in 1 mL of a mixture of chloroform and methanol (1:1).

Procedure—Apply separately 5-µL portions of the *Test solution* and the *Standard solution* to a suitable thin-layer chromatographic plate coated with a 0.25-mm layer of chromatographic silica gel mixture (see *Chromatography* ⟨621⟩). Allow the spots to dry, and develop the chromatogram in a solvent system consisting of a mixture of chloroform, methanol, and isopropylamine (92:3:3). Remove the plate from the developing chamber, mark the solvent front, and allow the solvent to evaporate. Spray the plate with *p*-nitrobenzenediazonium tetrafluoroborate solution, prepared by adding 250 mg to 5 mL of water, mixing, and filtering. Spray the plate with sodium carbonate solution (1 in 10): the R_F value of the principal spot obtained from the *Test solution* corresponds to that obtained from the *Standard solution.*

pH ⟨791⟩: between 5.0 and 7.5.

Assay—

Standard preparation—Dissolve an accurately weighed quantity of USP Xylometazoline Hydrochloride RS in water to obtain a solution having a known concentration of about 0.5 mg per mL. Transfer 10.0 mL of this solution to a 125-mL separator, and proceed as directed under *Assay preparation*, beginning with "add 10 mL each of water and dilute hydrochloric acid (1 in 6), respectively." The concentration of USP Xylometazoline Hydrochloride RS in the *Standard preparation* is about 100 µg per mL.

Assay preparation—Transfer an accurately measured volume of Nasal Solution, equivalent to about 5 mg of xylometazoline hydrochloride, to a 125-mL separator, add 10 mL each of water and dilute hydrochloric acid (1 in 6), respectively, and extract with three 10-mL portions of methylene chloride. Discard the methylene chloride extracts, add 10 mL of sodium hydroxide solution (1 in 5) to the separator, and extract with three 15-mL portions of methylene chloride. Filter the combined extracts through glass wool into a 50-mL volumetric flask, dilute with methylene chloride to volume, and mix.

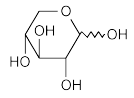
Procedure—Transfer 5.0 mL each of the *Standard preparation* and the *Assay preparation*, respectively, to separate 10-mL volumetric flasks, and evaporate in a water bath maintained at 40°, with the aid of a stream of nitrogen, to dryness. Dissolve the residue in each flask in 0.50 mL of dehydrated alcohol, and add 0.50 mL of dehydrated alcohol to a third 10-mL volumetric flask to provide the blank. To each flask add 0.50 mL of sodium hydroxide solution (1 in 25), swirl, to each add 5.0 mL of sodium nitroferricyanide solution (1 in 200), and mix. After 10 minutes, accurately timed, add 1.0 mL of a saturated solution of sodium bicarbonate to each flask, swirl, and allow to stand for 10 minutes. Dilute each with water to volume, mix, and allow to stand for 15 minutes. Concomitantly determine the absorbances of the solutions in 1-cm cells at the wavelength of maximum absorbance at about 565 nm, with a suitable spectrophotometer, using the blank to set the instrument. Calculate

the quantity, in mg, of xylometazoline hydrochloride ($C_{16}H_{24}N_2 \cdot HCl$) in each mL of the Nasal Solution taken by the formula:

$$(0.05C / V)(A_U / A_S)$$

in which C is the concentration, in µg per mL, of USP Xylometazoline Hydrochloride RS in the *Standard preparation, V* is the volume, in mL, of Nasal Solution taken, and A_U and A_S are the absorbances of the solutions from the *Assay preparation* and the *Standard preparation,* respectively.

Xylose



D-Xylopyranose

$C_5H_{10}O_5$ 150.13
D-Xylose.
D-Xylose [58-86-6; 6763-34-4].

» Xylose contains not less than 98.0 percent and not more than 102.0 percent of $C_5H_{10}O_5$, calculated on the dried basis.

Packaging and storage—Preserve in tight containers at controlled room temperature.

USP Reference standards ⟨11⟩**—**
USP Fructose RS
USP Xylose RS

Color of solution—A freshly prepared solution (1 in 10) is clear and colorless.

Identification—

A: *Infrared Absorption* ⟨197A⟩.

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

Specific rotation ⟨781S⟩: between +18.2° and +19.4°.

Test solution: 100 mg per mL, in 0.012 N ammonium hydroxide.

Loss on drying ⟨731⟩**—**Dry 2 g to 5 g at a pressure not exceeding 50 mm of mercury at 60° to constant weight, a current of dried air being passed through the oven during the drying period to remove water vapor: it loses not more than 0.1% of its weight.

Residue on ignition ⟨281⟩: not more than 0.05%.

Iron ⟨241⟩**—**Dissolve 2.0 g in 45 mL of water, and add 2 mL of hydrochloric acid: the limit is 5 ppm.

Heavy metals ⟨231⟩**—**Dissolve 2.0 g in water to make 25 mL of solution: the limit is 0.001%.

Chromatographic purity—Use the chromatogram of the *Assay preparation*, obtained as directed in the *Assay*. Calculate the percentage of each individual impurity, excluding any solvent peaks, in the portion of Xylose taken:

$$100(r_i / r_S)$$

in which r_i is the response of each individual impurity; and r_S is the sum of all of the responses in the chromatogram: not more than 1.0% of any individual impurity is found, and the sum of all individual impurities found is not more than 2.0%.

Assay—

Mobile phase—Use a degassed mixture of acetonitrile and water (75:25).

System suitability solution—Prepare a solution in *Mobile phase*, containing about 10 mg of USP Xylose RS and 0.2 mg of USP Fructose RS per mL.

Standard preparation—Dissolve an accurately weighed quantity of USP Xylose RS in *Mobile phase* to obtain a solution having a known concentration of about 10 mg per mL.

Assay preparation—Transfer about 100 mg of Xylose, accurately weighed, to a 10-mL volumetric flask, dissolve in and dilute with *Mobile phase* to volume, and mix.

Chromatographic system (see *Chromatography* ⟨621⟩)—The liquid chromatograph is equipped with a 192-nm detector and a 4.6-mm × 15-cm column that contains packing L8. The column temperature is maintained at 30°, and the flow rate is about 2 mL per minute. Chromatograph the *System suitability solution,* and record the peak responses as directed for *Procedure:* the resolution, *R,* between the xylose and fructose peaks is not less than 2.0. The relative standard deviation for replicate injections is not more than 2.0%.

Procedure—Separately inject equal volumes (about 25 µL) of the *Assay preparation* and the *Standard preparation* into the chromatograph, record the chromatograms, and measure the responses for the major peaks. Calculate the quantity, in mg, of $C_5H_{10}O_6$, in the portion of Xylose taken by the formula:

$$10C(r_U / r_S)$$

in which C is the concentration, in mg per mL, of USP Xylose 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.