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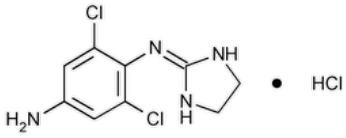
Apraclonidine Hydrochloride



$C_9H_{10}Cl_2N_4 \cdot HCl$ 281.57

1,4-Benzenediamine, 2,6-dichloro-N^1-2-imidazolidinylidene-, monohydrochloride.

2-[(4-Amino-2,6-dichlorophenyl)imino]imidazolidine monohydrochloride CAS RN®: 73218-79-8; UNII: D2VW67N38H.

» Apraclonidine Hydrochloride contains not less than 98.0 percent and not more than 102.0 percent of $C_9H_{10}Cl_2N_4 \cdot HCl$, calculated on the dried basis.

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

USP Reference standards ⟨11⟩—
USP Apraclonidine Hydrochloride RS

Identification—

Change to read:

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

B: It responds to the tests for *Chloride* ⟨191⟩.

pH ⟨791⟩: between 5.0 and 6.6 in a solution (1 in 100).

Loss on drying ⟨731⟩—Dry it in vacuum at 105° for 3 hours: it loses not more than 1.0% of its weight.

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

Chromatographic purity—

Phosphate buffer—Transfer 6.8 mL of phosphoric acid to a 2000-mL volumetric flask, add about 1900 mL of water, and mix. Adjust with sodium hydroxide solution (1 in 2) to a pH of 3.0, dilute with water to volume, and mix.

Mobile phase—Prepare a filtered and degassed mixture of acetonitrile, *Phosphate buffer*, and methanol (56:40:4). Make adjustments if necessary (see *System Suitability* under *Chromatography* ⟨621⟩).

System suitability solution—Prepare a solution in *Mobile phase* containing about 0.8 mg of USP Apraclonidine Hydrochloride RS per mL.

Test solution—Transfer about 20 mg of Apraclonidine Hydrochloride, accurately weighed, to a 25-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 220-nm detector and an 8-mm × 100-mm column that contains packing L7. The flow rate is about 3 mL per minute. Chromatograph the *System suitability solution*, and record the peak responses as directed for *Procedure:* the tailing factor for the apraclonidine peak is not more than 2.2, and the relative standard deviation for replicate injections is not more than 2.0%.

Procedure—Inject about 20 µL of the *Test solution* into the chromatograph, record the chromatogram, and measure the areas for the major peaks. [Note—Allow about five times the elution time of apraclonidine before making the next injection.] Calculate the percentage of each peak, other than the solvent peak and the apraclonidine peak, in the specimen of Apraclonidine Hydrochloride taken by the same formula:

$$100 r_i / r_t$$

in which r_i is the response of each peak other than the principal peak, and r_t is the sum of the responses of all of the peaks, excluding that of the solvent peak: not more than 1.0% for any individual impurity and not more than 2.0% total impurities are found.

Assay—Dissolve about 125 mg of Apraclonidine Hydrochloride, accurately weighed, in 40 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 from the second inflection point, using a calomel-glass electrode system (see *Titrimetry* ⟨541⟩). Perform a blank determination, and make any necessary correction. Each mL of 0.1 N perchloric acid is equivalent to 14.08 mg of $C_9H_{10}Cl_2N_4 \cdot HCl$.

Topic/Question	Contact	Expert Committee
APRACLONIDINE HYDROCHLORIDE	Documentary Standards Support	SM32020 Small Molecules 3

Chromatographic Database Information: Chromatographic Database

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

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