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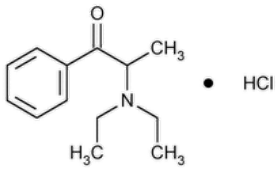
Diethylpropion Hydrochloride



$C_{13}H_{19}NO \cdot HCl$ 241.76

1-Propanone, 2-(diethylamino)-1-phenyl-, hydrochloride.

2-(Diethylamino)propiophenone hydrochloride CAS RN®: 134-80-5; UNII: 19V2PL39NG.

» Diethylpropion Hydrochloride contains not less than 97.0 percent and not more than 103.0 percent of $C_{13}H_{19}NO \cdot HCl$, calculated on the anhydrous basis. It may contain tartaric acid as a stabilizer.

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

Labeling—The label indicates whether it contains tartaric acid as a stabilizer.

USP Reference standards ⟨11⟩—
USP Diethylpropion Hydrochloride RS

Identification—

Change to read:

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

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

C: A solution (1 in 100) responds to the tests for *Chloride* ⟨191⟩.

Water Determination, *Method I* ⟨921⟩: not more than 0.5%.

Secondary amines—Dissolve 100 mg in 2 mL of methylene chloride in a centrifuge tube. Transfer to a second tube 2 mL of a Standard solution of diethylamine hydrochloride (dried at 105° for 2 hours before being used) in methylene chloride having a known concentration of 250 µg per mL. Treat each solution as follows. Extract with 2 mL of a buffer solution containing 5.7 g of sodium carbonate and 3.0 g of sodium bicarbonate per 100 mL of water. Centrifuge, if necessary, to clarify the upper phase, and immediately transfer 0.5 mL of it to a spot plate. Immediately add 2 drops of acetaldehyde TS, and then, in rapid succession, add 1 drop of sodium nitroferricyanide solution (1 in 100) to each spot. Immediately and simultaneously stir both spots to mix the reagents: any blue color produced within 3 minutes by the test solution is not more intense than that of the Standard solution (not more than 0.5% of secondary amines as diethylamine hydrochloride).

Free bromine—One drop of a solution (1 in 10) produces no discoloration when placed upon starch iodide paper.

Limit of hydrobromic acid and bromide—To 10 mL of a solution (1 in 10) add 1 mL of sodium hydroxide solution (1 in 10), extract with about 25 mL of chloroform, and discard the chloroform extract. Add 1 mL of 6 N hydrochloric acid, 0.5 mL of chloroform, and 0.5 mL of freshly prepared chloramine T solution (1 in 10), and shake vigorously: no yellow or brown-red color is produced in the chloroform layer.

Chromatographic purity—

Phosphate buffer—Dissolve 136.1 g of monobasic potassium phosphate in 900 mL of water, add 3.2 mL of phosphoric acid, dilute with water to 1000 mL, and mix.

Diluent—Prepare a mixture of water, *Phosphate buffer*, and acetonitrile (8:1:1).

Mobile phase—Mix 100 mL of acetonitrile, 100 mL of *Phosphate buffer*, 7.0 mL of diethylamine, and sufficient water to make 1 L. Filter, and degas before use. Make adjustments if necessary (see *System Suitability* under *Chromatography* ⟨621⟩).

Test preparation—Transfer 100 mg of Diethylpropion Hydrochloride, accurately weighed, to a 50-mL volumetric flask, dissolve in about 40 mL of *Diluent*, add *Diluent* to volume, and mix.

Standard preparation—Dissolve an accurately weighed quantity of USP Diethylpropion Hydrochloride RS in *Diluent*, and dilute quantitatively, and stepwise if necessary, with *Diluent* to obtain a solution having a known concentration of about 0.01 mg per mL.

System suitability solution—Prepare a solution in *Diluent* containing about 25 µg of 2-ethylaminopropiophenone hydrochloride and 50 µg of USP Diethylpropion Hydrochloride RS per mL.

Chromatographic system (see *Chromatography* ⟨621⟩)—The liquid chromatograph is equipped with a 254-nm detector and a 4.6-mm × 15-cm column that contains packing L11. The flow rate is about 1 mL per minute. Chromatograph the *System suitability solution*, and record the peak responses as directed for *Procedure:* the relative retention times are about 0.5 for 2-ethylaminopropiophenone and 1.0 for diethylpropion, and

the resolution, *R,* between the 2-ethylaminopropiophenone and diethylpropion peaks is not less than 6.0. Chromatograph the *Standard preparation*, and record the peak responses as directed for *Procedure:* the relative standard deviation for replicate injections is not more than 1.0%.

Procedure—Separately inject equal volumes (about 20 µL) of the *Standard preparation* and the *Test preparation* into the chromatograph, record the chromatograms, and measure the peak responses. The sum of all of the peak responses, excluding the solvent peak responses and the diethylpropion response, from the *Test preparation* is not greater than the diethylpropion response from the *Standard preparation* (0.5%).

Assay—

Phosphate buffer—Dissolve 136.1 g of monobasic potassium phosphate in 900 mL of water, add 4.3 mL of phosphoric acid, dilute with water to 1000 mL, and mix.

Mobile phase—Prepare a suitable mixture of water, acetonitrile, *Phosphate buffer*, and 1.0 M sodium nitrate (730:200: 50:20), filter through a membrane filter (0.7-µm or finer porosity), and degas. Make adjustments if necessary (see *System Suitability* under *Chromatography ⟨621⟩*).

Standard preparation—Dissolve an accurately weighed quantity of USP Diethylpropion Hydrochloride RS in *Mobile phase*, and dilute quantitatively, and stepwise if necessary, with *Mobile phase* to obtain a solution having a known concentration of about 40 µg per mL.

Assay preparation—Transfer about 100 mg of Diethylpropion Hydrochloride, accurately weighed, to a 250-mL volumetric flask, dissolve in *Mobile phase*, dilute with *Mobile phase* to volume, and mix. Transfer 10.0 mL of this solution to a 100-mL volumetric flask, dilute with *Mobile phase* to volume, and mix.

System suitability preparation—Prepare a solution in *Mobile phase* containing about 200 µg of benzoic acid and 40 µg of USP Diethylpropion Hydrochloride RS per mL.

Chromatographic system (see *Chromatography ⟨621⟩*)—The liquid chromatograph is equipped with a 254-nm detector and a 4-mm × 30-cm column that contains packing L1. The flow rate is about 1.5 mL per minute. Chromatograph the *System suitability preparation*, and record the peak responses as directed for *Procedure:* the relative retention times are about 0.5 for diethylpropion hydrochloride and 1.0 for benzoic acid, and the resolution, *R,* between the diethylpropion hydrochloride and benzoic acid peaks is not less than 2.0. Chromatograph the *Standard preparation*, and record the peak responses as directed for *Procedure:* the relative standard deviation for replicate injections is not more than 1.0%.

Procedure—Separately inject equal volumes (about 50 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 $C_{13}H_{19}NO \cdot HCl$ in the portion of Diethylpropion Hydrochloride taken by the formula:

$$2.5C(r_U/r_S)$$

in which C is the concentration, in µg per mL, of USP Diethylpropion Hydrochloride 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
DIETHYLPROPION HYDROCHLORIDE	Documentary Standards Support	SM22020 Small Molecules 2

Chromatographic Database Information: Chromatographic Database

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

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