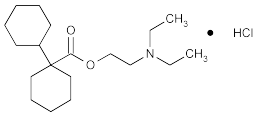
[NOTE—Use this *Assay preparation* promptly, or refrigerate and use on the day prepared.]

Procedure—Proceed as directed for *Procedure* in the *Assay* under *Dicloxacillin Sodium*. Calculate the quantity, in mg, of dicloxacillin ($C_{19}H_{17}Cl_2N_3O_3S$) in each mL of the constituted Dicloxacillin for Oral Suspension taken by the formula:

$$(125 + V)(CE / 1000V)(r_U / r_S)$$

in which V is the volume, in mL, of constituted Dicloxacillin for Oral Suspension taken to prepare the *Assay preparation;* and the other terms are as defined therein.

Dicyclomine Hydrochloride



$C_{19}H_{35}NO_2 \cdot HCl$ 345.95

[Bicyclohexyl]-1-carboxylic acid, 2-(diethylamino)ethyl ester, hydrochloride.

2-(Diethylamino)ethyl[bicyclohexyl]-1-carboxylate hydrochloride [67-92-5].

» Dicyclomine Hydrochloride contains not less than 99.0 percent and not more than 102.0 percent of $C_{19}H_{35}NO_2 \cdot HCl$, calculated on the dried basis.

Packaging and storage—Preserve in well-closed containers.

USP Reference standards ⟨11⟩**—**
USP Dicyclomine Hydrochloride RS

Identification—

A: *Infrared Absorption* ⟨197K⟩.

B: Mix about 5 mL of a 1 in 500 solution of it with about 2 mL of 2 N nitric acid, and add about 2 mL of silver nitrate TS: a white precipitate is formed which is insoluble in nitric acid but soluble in a slight excess of 6 N ammonium hydroxide.

Melting range, *Class I* ⟨741⟩: between 169° and 174°.

pH ⟨791⟩: between 5.0 and 5.5, in a solution (1 in 100).

Loss on drying ⟨731⟩**—**Dry it at 105° for 4 hours: it loses not more than 1.0% of its weight.

Readily carbonizable substances ⟨271⟩**—**Dissolve 500 mg in 5 mL of sulfuric acid: the solution has no more color than *Matching Fluid D.*

Assay—Dissolve about 600 mg of Dicyclomine Hydrochloride, accurately weighed, in 70 mL of glacial acetic acid, add 10 mL of mercuric acetate TS and 1 drop of crystal violet TS, and titrate with 0.1 N perchloric acid VS to a blue endpoint. Perform a blank determination, and make any necessary correction. Each mL of 0.1 N perchloric acid is equivalent to 34.60 mg of $C_{19}H_{35}NO_2 \cdot HCl$.

Dicyclomine Hydrochloride Capsules

» Dicyclomine Hydrochloride Capsules contain not less than 93.0 per cent and not more than 107.0 percent of the labeled amount of dicyclomine hydrochloride ($C_{19}H_{35}NO_2 \cdot HCl$).

Packaging and storage—Preserve in well-closed containers.

USP Reference standards ⟨11⟩**—**
USP Dicyclomine Hydrochloride RS

Identification—

A: Transfer a portion of the contents of the Capsules, equivalent to about 100 mg of dicyclomine hydrochloride, to a separator containing 10 mL of water and 1 mL of hydrochloric acid. Extract the aqueous acid solution with two 30-mL portions of chloroform, transfer the chloroform extracts to a second separator containing 20 mL of water and 1 mL of sodium hydroxide solution (1 in 10), and shake. Filter the chloroform layer through anhydrous sodium sulfate into a suitable container, and add 3 mL of a freshly prepared 1 in 20 solution of acetyl chloride in anhydrous methanol, prepared by cautiously adding acetyl chloride dropwise to anhydrous methanol with stirring. Evaporate under reduced pressure at room temperature until the residue has been thoroughly dried: the IR absorption spectrum of a potassium bromide dispersion of the dicyclomine hydrochloride so obtained exhibits maxima and minima at the same wavelengths as that of a similar preparation of USP Dicyclomine Hydrochloride RS.

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.*

Dissolution ⟨711⟩**—**

Medium: 0.01 N hydrochloric acid; 500 mL.

Apparatus 2: 50 rpm.

Time: 45 minutes.

Determine the amount of $C_{19}H_{35}NO_2 \cdot HCl$ dissolved by employing the following method.

Mobile phase—Prepare as directed in the *Assay.*

0.04 M Phosphate buffer, pH 7.5—Dissolve 2.72 g of monobasic potassium phosphate in 450 mL of water, adjust with 10% sodium hydroxide to a pH of 7.5 ± 0.1, dilute with water to 500 mL, and mix.

Buffer–acetonitrile mixture—Prepare a mixture of *0.04 M Phosphate buffer, pH 7.5* and acetonitrile (1:1).

Standard solution—Prepare a solution in *Medium* having a known concentration of about 20 µg per mL of USP Dicyclomine Hydrochloride RS. Transfer 25.0 mL of this solution to a suitable flask, add 25.0 mL of the *Buffer–acetonitrile mixture,* and mix.

Test solution—Pass a portion of the solution under test through a 0.7-µm glass microfiber filter. Transfer 5.0 mL of the filtrate to a suitable flask, add 5.0 mL of the *Buffer–acetonitrile mixture,* and mix.

Chromatographic system (see *Chromatography* ⟨621⟩)—The liquid chromatograph is equipped with a 215-nm detector and a 4.6-mm × 15-cm column containing 3.5-µm packing L7. The flow rate is about 1.0 mL per minute. Chromatograph the *Standard solution,* and record the responses as directed for *Procedure:* the tailing factor for the analyte peak is not more than 2.0, and the relative standard deviation for replicate injections is not more than 2.0%.

Procedure—Separately inject equal volumes (about 250 µL) of the *Standard solution* and the *Test solution* into the chromatograph, record the chromatograms, and measure the responses for the major peaks. Calculate the amount, in mg, of $C_{19}H_{35}NO_2 \cdot HCl$ dissolved.

Tolerances—Not less than 75% *(Q)* of the labeled amount of $C_{19}H_{35}NO_2 \cdot HCl$ is dissolved in 45 minutes.

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

Assay—

0.02 M Phosphate buffer, pH 7.5—Dissolve 2.72 g of monobasic potassium phosphate in 900 mL of water, adjust with 10% sodium hydroxide to a pH of 7.5 ± 0.1, dilute with water to 1000 mL, and mix.

Mobile phase—Prepare a mixture of acetonitrile and *0.02 M Phosphate buffer, pH 7.5* (70:30), filter, and degas. Make adjust-