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Methyclothiazide Tablets

» Methyclothiazide Tablets contain not less than 90.0 percent and not more than 110.0 percent of the labeled amount of methyclothiazide ($C_9H_{11}Cl_2N_3O_4S_2$).

Packaging and storage—Preserve in well-closed containers.

USP Reference standards ⟨11⟩—
USP Methyclothiazide RS

Change to read:

Identification, ▲**Spectroscopic Identification Tests** ⟨197⟩, *Ultraviolet-Visible Spectroscopy*: **197U**▲ (CN 1-May-2020) —

Solution—Powder a number of Tablets, equivalent to about 50 mg of methyclothiazide, and transfer to a 100-mL volumetric flask with the aid of methanol. Add about 60 mL of methanol, and shake the flask for 1 hour. Dilute with methanol to volume, mix, and centrifuge a portion of the solution. Pipet 2 mL of the clear supernatant into a second 100-mL volumetric flask, dilute with methanol to volume, and mix: the UV absorption spectrum of this solution exhibits maxima and minima only at the same wavelengths as that of a similar solution of USP Methyclothiazide RS.

Dissolution ⟨711⟩—
Medium: 0.01 N hydrochloric acid; 900 mL.
Apparatus 2: 50 rpm.
Time: 60 minutes.
Procedure—Determine the amount of $C_9H_{11}Cl_2N_3O_4S_2$ dissolved by employing UV absorption at the wavelength of maximum absorbance at about 270 nm on filtered portions of the solution under test, suitably diluted with *Dissolution Medium*, if necessary, in comparison with a Standard solution having a known concentration of USP Methyclothiazide RS in the same *Medium*. An amount of alcohol not to exceed 1% of the total volume of the Standard solution may be used to dissolve USP Methyclothiazide RS prior to dilution with *Dissolution Medium*.
Tolerances—Not less than 70% *(Q)* of the labeled amount of $C_9H_{11}Cl_2N_3O_4S_2$ is dissolved in 60 minutes.

Uniformity of dosage units ⟨905⟩: meet the requirements.
Procedure for content uniformity—Transfer 1 finely powdered Tablet to a 50-mL volumetric flask, add about 30 mL of methanol, and shake by mechanical means for 1 hour. Dilute with methanol to volume, mix, and centrifuge a portion of the mixture. Dilute quantitatively with methanol to obtain a solution containing approximately 10 µg per mL of methyclothiazide. Concomitantly determine the absorbances of this solution and a Standard solution of USP Methyclothiazide RS in the same medium, having a known concentration of about 10 µg per mL, in 1-cm cells at the wavelength of maximum absorbance at about 267 nm, with a suitable spectrophotometer, using methanol as the blank. Calculate the quantity, in mg, of $C_9H_{11}Cl_2N_3O_4S_2$ in the Tablet taken by the formula:

$$(TC/D)(A_U/A_S)$$

in which T is the labeled quantity, in mg, of methyclothiazide in the Tablet; C is the concentration, in µg per mL, of USP Methyclothiazide RS in the Standard solution; D is the concentration, in µg per mL, of methyclothiazide in the solution from the Tablet, based upon the labeled quantity per Tablet and the extent of dilution; and A_U and A_S are the absorbances of the solution from the Tablet and the Standard solution, respectively.

Assay—
Standard preparation—Transfer about 20 mg of USP Methyclothiazide RS, accurately weighed, to a 100-mL volumetric flask, add methanol to volume, and mix. Transfer 10.0 mL of this solution to a 200-mL volumetric flask, add chloroform to volume, and mix.
Assay preparation—Weigh and finely powder not fewer than 20 Tablets. Transfer an accurately weighed portion of the powder, equivalent to about 2 mg of methyclothiazide, to a 150-mL beaker, add 2.0 mL of methanol, mix, allow the mixture to stand for 30 minutes while taking precautions against loss of solvent, add 2.0 mL of 0.1 M sodium bicarbonate, and mix.
Procedure—[Note—Use water-saturated solvents throughout this procedure.] Mix about 3 g of chromatographic siliceous earth with 2.0 mL of 0.1 M sodium bicarbonate in a 150-mL beaker. Pack the mixture into a 25- × 200-mm chromatographic column. Add 4 g of chromatographic siliceous earth to the *Assay preparation*, mix, transfer the mixture to the column, and pack. Dry-wash the beaker with 1 g of the siliceous earth mixed with 3 drops of water, and transfer to the column. Place a small pad of glass wool above the column packing, pass 75 mL of a mixture of isooctane and ether (9:1) through the column, and discard the eluate. Using a 200-mL volumetric flask as a receiver, pass 100 mL of chloroform through the column, wash the tip of column with ether, add 10.0 mL of methanol, dilute with chloroform to volume, and mix. Concomitantly determine the absorbances of this solution and the *Standard preparation* at the wavelength of maximum absorbance at about

268 nm, with a suitable spectrophotometer, using chloroform as the blank. Calculate the quantity, in mg, of methyclothiazide ($C_9H_{11}Cl_2N_3O_4S_2$) in the portion of Tablets taken by the formula:

$$0.2C(A_U/A_S)$$

in which C is the concentration, in µg per mL, of USP Methyclothiazide RS in the *Standard preparation;* and A_U and A_S are the absorbances of the solution 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
METHYCLOTHIAZIDE TABLETS	Documentary Standards Support	SM22020 Small Molecules 2

Chromatographic Database Information: Chromatographic Database

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