Medroxyprogesterone / Official Monographs

bring the Reference Standard into solution prior to dilution with water. Prepare this Standard stock solution fresh daily.

Standard solution—Pipet a 20-mL aliquot of Standard stock solution into a 1 L volumetric flask. Add 40 mL of Sodium lauryl sulfate stock solution, and dilute with water to volume. This solution is stable for up to 7 days.

Test solution—Withdraw 15 mL of the solution under test, and filter, discarding the first 5 mL of the filtrate.

Mobile phase—Prepare a filtered and degassed solution of acetonitrile and water (60:40). Make adjustments if necessary (see System Suitability under Chromatography (621)).

Chromatographic system (see Chromatography (621))—The liquid chromatograph is equipped with a 254-nm detector and a 4-mm × 8-cm column that contains packing L7. The flow rate is about 1.5 mL per minute. Chromatograph the Standard solution, and record the peak responses as directed for Procedure: the tailing factor for the analyte peak is not more than 1.2; and the relative standard deviation for replicate injections is not more than 2.0%.

Procedure—Separately inject equal volumes (about 20 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 percentage of C_{24}H_{34}O_4 in the peak responses so obtained.

Tolerances—Not less than 50% (Q) of the labeled amount of C_{24}H_{34}O_4 is dissolved in 45 minutes.

Uniformity of dosage units (905): meet the requirements.

Procedure for content uniformity—Dissolve an accurately weighed portion of USP Medroxyprogesterone Acetate RS in a mixture of alcohol and water (3:1) to obtain a solution having a known concentration of about 15 µg per mL. Transfer 1 Tablet to a volumetric flask, add a mixture of alcohol and water (3:1) to volume, and shake for about 15 minutes. Filter, and quantitatively and stepwise if necessary, with Mobile phase to obtain a final solution containing about 15 µg per mL. Concomitantly determine the absorbances of this solution and the Standard solution in 1-cm cells at the wavelength of maximum absorbance at about 242 nm. Calculate the quantity, in mg, of C_{24}H_{34}O_4 in the Tablet taken by the formula:

\[
\frac{T(D)}{D} = \frac{C}{A_0} = A_1
\]

in which \( T \) is the labeled quantity, in mg, of medroxyprogesterone acetate in the Tablet; \( D \) is the concentration, in µg per mL, of medroxyprogesterone acetate in the solution from the Tablet; \( C \) is the concentration, in µg per mL, of USP Medroxyprogesterone Acetate RS in the Standard solution, and \( A_0 \) and \( A_1 \) are the absorbances of the solution from the Tablet and the Standard solution, respectively.

Assay—

Mobile phase, Standard preparation, and Chromatographic system—Prepare as directed in the Assay under Medroxyprogesterone Acetate.

Assay preparation—Weigh finely powder not fewer than 20 Tablets. Weigh accurately a portion of the powder, equivalent to about 25 mg of medroxyprogesterone acetate, into a 50-mL glass centrifuge tube. Pipet 25 mL of acetonitrile into the tube, shake to wet the powder thoroughly, and sonicate for not less than 10 minutes, and centrifuge. Use the clear supernatant as the Assay preparation.

Procedure—Proceed as directed for Procedure in the Assay under Medroxyprogesterone Acetate. Calculate the quantity, in mg, of medroxyprogesterone acetate (C_{24}H_{34}O_4) in the portion of Tablets taken by the formula:

\[
25C(r_i / r_f)
\]

in which \( C \) is the concentration, in mg per mL, of USP Medroxyprogesterone Acetate RS in the Standard preparation; and \( r_i \) and \( r_f \) are the peak responses obtained from the Assay preparation and the Standard preparation, respectively.

Mefenamic Acid

C_{13}H_{13}NO_2  241.29
Benzonic acid, 2-(2,3-dimethylphenyl)aminoo- N-2,3-Xylylanthranilic acid  [61-68-7].

» Mefenamic Acid contains not less than 98.0 percent and not more than 102.0 percent of C_{13}H_{13}NO_2, calculated on the dried basis.

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

USP Reference standards (11)—

USP Mefenamic Acid RS

Identification—

A: Infrared Absorption (197K).
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.

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

Residue on ignition (281): not more than 0.1%.

Heavy metals, Method II (231): 0.002%.

Chromatographic purity—

Buffer solution, Mobile phase, and Chromatographic system—Proceed as directed in the Assay.

Standard solution—Dissolve an accurately weighed quantity of USP Mefenamic Acid RS in Mobile phase to obtain a solution having a known concentration of about 10 µg per mL.

Test solution—Transfer about 100 mg of Mefenamic Acid, accurately weighed, to a 100-mL volumetric flask, dissolve in and dilute with Mobile phase to volume, and mix.

Procedure—Separately inject equal volumes (about 10 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 percentage of each impurity in the portion of Mefenamic Acid taken by the formula:

\[
100(C_i / C_0)(r_i / r_f)
\]

in which \( C_i \) is the concentration, in µg per mL, of USP Mefenamic Acid RS in the Standard solution; \( C_0 \) is the concentration, in µg per mL, of Mefenamic Acid in the Test solution; \( r_i \) is the peak response for each impurity obtained from the Test solution; and \( r_f \) is the peak response for mefenamic acid obtained from the Standard solution: not more than 0.1% of any individual impurity is found; and not more than 0.5% of total impurities is found.

Assay—

Buffer solution—Prepare a 50 mM solution of monobasic ammonium phosphate, and adjust with 3 M ammonium hydroxide to a pH of 5.0.

Mobile phase—Prepare a filtered and degassed mixture of acetonitrile, Buffer solution, and tetrahydrofuran (23:20:7). Make adjustments if necessary (see System Suitability under Chromatography (621)).

Standard preparation—Dissolve an accurately weighed quantity of USP Mefenamic Acid RS in Mobile phase, and dilute quantitatively, and stepwise if necessary, with Mobile phase to obtain a solution having a known concentration of about 0.2 mg per mL.

Assay preparation—Transfer about 100 mg of Mefenamic Acid, accurately weighed, to a 500-mL volumetric flask, dissolve in and dilute with Mobile phase to volume, and mix.
Mefenamic Acid Capsules

» Mefenamic Acid Capsules contain not less than 90.0 percent and not more than 110.0 percent of the labeled amount of mafenamic acid (C₁₅H₁₅NO₂).

Packaging and storage—Preserve in tight containers.

USP Reference standards (11)—USP Mefenamic Acid RS

Identification—

A: Place a portion of Capsule contents, equivalent to about 250 mg of mafenamic acid, in a 250-mL volumetric flask, add about 100 mL of a mixture of chloroform and methanol (3:1), and shake vigorously. Dilute with a mixture of chloroform and methanol (3:1) to volume, mix, and filter. The filtrate so obtained responds to the Thin-layer Chromatographic Identification Test (201), a solvent system consisting of a mixture of chloroform, ethyl acetate, and glacial acetic acid (75:25:1) and the Ordinance Impurities (466) visualization technique 17 being used.

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

Dissolution (711)—

0.05 M Tris buffer—Dissolve 60.5 g of tris(hydroxymethyl)aminomethane in 6 L of water, and dilute with water to 10 L. Adjust with phosphoric acid to a pH of 9.0 ± 0.05. To a second container, transfer about 6 liters of this solution, add 100 g of methanol (3:1) to volume, mix, and filter: the filtrate so obtained responds to the Thin-layer Chromatographic Identification Test (201), a solvent system consisting of a mixture of chloroform, ethyl acetate, and glacial acetic acid (75:25:1) and the Ordinance Impurities (466) visualization technique 17 being used.

Tolerances—Not less than 75% (Q) of the labeled amount of C₁₅H₁₅NO₂ is dissolved in 45 minutes.

Uniformity of dosage units (905): meet the requirements.

Assay—

Mobile phase, Standard preparation, and Chromatographic system—Proceed as directed in the Assay under Mefenamic Acid.

Assay preparation—Remove, as completely as possible, the contents of not fewer than 20 Capsules. Weigh the contents, and determine the average weight per capsule. Mix the combined contents, and transfer an accurately weighed quantity of the powder, equivalent to about 100 mg of mafenamic acid, to a 500-mL volumetric flask. Add 10.0 mL of tetrahydrofuran, and sonicate for about 5 minutes with occasional mixing. Dilute with Mobile phase to volume, mix, and filter.

Procedure—Proceed as directed for Procedure in the Assay under Mefenamic Acid. Calculate the quantity, in mg, of C₁₅H₁₅NO₂ in the portion of Capsules taken by the formula:

\[
500C(r_0 / r_i)
\]

in which C is the concentration, in mg per mL, of USP Mefenamic Acid RS in the Standard preparation; and \(r_0\) and \(r_i\) are the mafenamic acid peak responses obtained from the Assay preparation and the Standard preparation, respectively.

Mefloquine Hydrochloride

C₁₅H₁₅NO₂ · HCl 414.77

4-Quinolinemethanol, α-2-piperidinyl-2,8-bis(trifluoromethyl)-, monohydrochloride, (R, S)-; D-erythro-α-2-Piperidyl-2,8-bis(trifluoromethyl)-4-quinolinemethanol monohydrochloride [51773-92-3].

DEFINITION

Mefloquine Hydrochloride contains NLT 98.0% and NMT 102.0% of C₁₇H₁₆F₆N₂O · HCl, calculated on the anhydrous basis.

IDENTIFICATION

A. INFRARED ABSORPTION (197K)

B. IDENTIFICATION TESTS—GENERAL, Chloride (191)

ASSAY—

PROCEDURE

Solution A: 1.5 g/L of sodium hydrogen sulfate in water

Mobile phase: Dissolve 1 g of tetrahepsyramonium bromide in a 1000-mL mixture of acetonitrile, methanol, and Solution A (2:1:2).

System suitability solution: 4 μg/mL each of USP Mefloquine Hydrochloride RS and USP Mefloquine Related Compound A RS in Mobile phase

Standard solution: 0.2 mg/mL of USP Mefloquine Hydrochloride RS in Mobile phase

Sample solution: 0.2 mg/mL of Mefloquine Hydrochloride in Mobile phase

Chromatographic system

(See Chromatography (621), System Suitability.)

Mode: LC

Detector: UV 280 nm

Guard column: 4-mm × 3-cm; C18 (recommended)

Column: 4.0-mm × 25-cm; 5-μm packing L1

Column temperature: 25°C

Flow rate: 0.8 mL/min

Injection size: 20 μL

System suitability—

Samples: System suitability solution and Standard solution

NOTES—The relative retention times for mefloquine related compound A and mefloquine are about 0.7 and 1.0, respectively.

Suitability requirements

Resolution: NLT 2.0 between mefloquine related compound A and mefloquine, System suitability solution