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Methoxsalen Capsules

DEFINITION

Methoxsalen Capsules contain NLT 90.0% and NMT 110.0% of the labeled amount of methoxsalen ($C_{12}H_8O_4$).

IDENTIFICATION

- **A.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the Assay.

Change to read:

- **B.** ▲▲ [SPECTROSCOPIC IDENTIFICATION TESTS \(197\)](#), [Ultraviolet-Visible Spectroscopy: 197U](#) ▲ (CN 1-MAY-2020) ▲ (USP 1-MAY-2019)

Sample solution: Nominally 4 µg/mL of methoxsalen in [alcohol](#) prepared as follows. Place 1 Capsule in 50 mL of [alcohol](#) contained in a high-speed glass blender jar, blend thoroughly until the shell is completely dispersed, and dilute a portion with [alcohol](#).

Acceptance criteria: The UV absorption spectrum of the *Sample solution* exhibits maxima and minima at the same wavelengths as those of a similar solution of [USP Methoxsalen RS](#), concomitantly measured.

▲[NOTE—The UV spectra of the major peaks of the *Sample solution* and the *Standard solution* as obtained in the Assay may also be used to meet the *Acceptance criteria*.]▲ (USP 1-MAY-2019)

ASSAY

Change to read:

- **PROCEDURE**

Mobile phase: [Acetonitrile](#) and [water](#) (65:35)

Standard stock solution: 0.2 mg/mL of [USP Methoxsalen RS](#) in [alcohol](#)

Standard solution: 4 µg/mL of [USP Methoxsalen RS](#) in *Mobile phase*, from the *Standard stock solution*

Sample stock solution 1 (for hard gelatin Capsules): Nominally equivalent to 0.04 mg/mL of methoxsalen in [alcohol](#) prepared as follows. Place NLT 10 Capsules in a high-speed glass blender jar containing 100.0 mL of [alcohol](#), and blend thoroughly. Transfer a portion of the aliquot, nominally equivalent to 2 mg of methoxsalen, from the blender jar into a 50-mL volumetric flask, dilute with [alcohol](#) to volume, and filter.

Sample stock solution 2 (for soft gelatin Capsules): Nominally equivalent to 0.04 mg/mL of methoxsalen in [alcohol](#) prepared as follows. Place the end of a long-stem glass funnel on a 250-mL volumetric flask, punch a hole at each end of a Capsule with a syringe containing 15 mL of [alcohol](#), and rinse the contents into the flask. Cut the Capsule shell with a scalpel, and wash the inside of the shell with 15 mL of [alcohol](#) into the same flask. Repeat these steps for NLT 4 additional Capsules, and collect the rinsings. Wash the funnel, and collect the rinsings in the same flask. Dilute with [alcohol](#) to volume, and mix. Transfer a portion of this solution, nominally equivalent to 2 mg of methoxsalen, into a 50-mL volumetric flask. Dilute with [alcohol](#) to volume, and filter.

Sample solution: Nominally equivalent to 4 µg/mL of methoxsalen in *Mobile phase*, from *Sample stock solution 1* or *Sample stock solution 2*

Chromatographic system

(See [Chromatography \(621\)](#), [System Suitability](#).)

Mode: LC

Detector: UV 254 nm. ▲For *Identification B*, use a diode array detector set at 200–400 nm.▲ (USP 1-May-2019)

Column: 4-mm × 30-cm; packing [L1](#)

Flow rate: 1.5 mL/min

Injection volume: 20 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Relative standard deviation: NMT 2.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of methoxsalen ($C_{12}H_8O_4$) in the portion of Capsules taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak response of methoxsalen from the *Sample solution*

r_s = peak response of methoxsalen from the *Standard solution*

C_s = concentration of [USP Methoxsalen RS](#) in the *Standard solution* (mg/mL)

C_u = nominal concentration of methoxsalen in the *Sample solution* (mg/mL)

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

Change to read:

- [DISSOLUTION \(711\)](#).

For soft gelatin Capsules

Medium: [Water](#); 900 mL

Apparatus 2: 50 rpm ▲with sinkers, if necessary¹▲ (USP 1-May-2019)

Time: 45 min

Standard solution: [USP Methoxsalen RS](#) in *Medium*. An amount of alcohol NMT 1% of the total volume of the *Standard solution* may be used to bring the Reference Standard into solution before diluting with *Medium*.

Sample solution: Dilute a filtered portion of the solution under test with *Medium* to a concentration similar to that of the *Standard solution*.

Instrumental conditions

Mode: UV

Analytical wavelength: Maximum absorbance at about 300 nm

Analysis

Samples: *Standard solution* and *Sample solution*

▲Calculate the percentage of the labeled amount of methoxsalen ($C_{12}H_8O_4$) dissolved:

$$\text{Result} = (A_u/A_s) \times C_s \times V \times D \times (1/L) \times 100$$

A_u = absorbance of the *Sample solution*

A_s = absorbance of the *Standard solution*

C_s = concentration [USP Methoxsalen RS](#) in the *Standard solution* (mg/mL)

V = volume of *Medium*, 900 mL

D = dilution factor for the *Sample solution*, if needed

L = label claim (mg/Capsule)▲ (USP 1-May-2019)

Tolerances: NLT 75% (Q) of the labeled amount of methoxsalen ($C_{12}H_8O_4$) is dissolved.

For hard gelatin Capsules

Medium: [Water](#); 900 mL

Apparatus 1: 150 rpm

Time: 90 min

Sample solution: Filter a portion of the solution under test.

Standard solution: [USP Methoxsalen RS](#) in [alcohol](#) and diluted with *Medium* to a concentration similar to that of the *Sample solution*

Instrumental conditions

Mode: UV

Analytical wavelength: Maximum absorbance at about 252 nm

Analysis

Samples: *Sample solution* and *Standard solution*

▲Calculate the percentage of the labeled amount of methoxsalen ($C_{12}H_8O_4$) dissolved:

$$\text{Result} = (A_u/A_s) \times C_s \times V \times (1/L) \times 100$$

A_u = absorbance of the *Sample solution*

A_s = absorbance of the *Standard solution*

C_s = concentration of [USP Methoxsalen RS](#) in the *Standard solution* (mg/mL)

V = volume of *Medium*, 900 mL

L = label claim (mg/Capsule)▲ (USP 1-May-2019)

Tolerances: NLT 75% (Q) of the labeled amount of methoxsalen ($C_{12}H_8O_4$) is dissolved.