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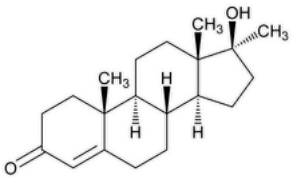
Methyltestosterone

Change to read:



$C_{20}H_{30}O_2$ ▲302.46▲ (USP 1-May-2024)

Androst-4-en-3-one, 17-hydroxy-17-methyl,(17β)-;

17β-Hydroxy-17-methylandrost-4-en-3-one CAS RN®: 58-18-4; UNII: V9EFU16ZIF.

DEFINITION

Methyltestosterone contains NLT 97.0% and NMT 103.0% of methyltestosterone ($C_{20}H_{30}O_2$), calculated on the dried basis.

IDENTIFICATION

Change to read:

• **A. Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*:** ▲197A or▲ (USP 1-May-2024) 197K

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

ASSAY

Change to read:

• **Procedure**

▲[Note—Protect solutions containing methyltestosterone from light.]▲ (USP 1-May-2024)

Mobile phase: Acetonitrile and water (55:45)

Standard stock solution: 0.25 mg/mL of USP Methyltestosterone RS in methanol

Standard solution: 0.02 mg/mL of USP Methyltestosterone RS from the *Standard stock solution* in *Mobile phase*

System suitability stock solution: 0.25 mg/mL of USP Testosterone RS in methanol

System suitability solution: ▲0.02 mg/mL each of USP Methyltestosterone RS and USP Testosterone RS in *Mobile phase* prepared as follows.▲ (USP 1-May-2024) Dilute 4 mL of the *System suitability stock solution* with the *Standard solution* to 50 mL.

Sample stock solution: 0.50 mg/mL of Methyltestosterone in methanol

Sample solution: 0.02 mg/mL of Methyltestosterone from the *Sample stock solution* in *Mobile phase*

Chromatographic system

(See *Chromatography ⟨621⟩, System Suitability*.)

Mode: LC

Detector: UV 241 nm

Column: 4-mm × 25-cm; ▲5-µm▲ (USP 1-May-2024) packing L1

Flow rate: 1 mL/min

Injection volume: 50 µL

System suitability

Samples: *Standard solution* and *System suitability solution*

[Note—The relative retention times for testosterone and methyltestosterone are about 0.8 and 1.0, respectively.]

Suitability requirements

Resolution: NLT 2.0 between testosterone and methyltestosterone, *System suitability solution*

▲▲ (USP 1-May-2024)

Tailing factor: NMT 2.7, *Standard solution*

Relative standard deviation: NMT ▲1.10%▲ (USP 1-May-2024) *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of methyltestosterone ($C_{20}H_{30}O_2$) in the portion of Methyltestosterone taken:

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

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

r_S = peak response of methyltestosterone from the *Standard solution*

C_S = concentration of USP Methyltestosterone RS in the *Standard solution* (mg/mL)

C_U = concentration of Methyltestosterone in the *Sample solution* (mg/mL)

Acceptance criteria: 97.0%–103.0% on the dried basis

IMPURITIES

Change to read:

• **Organic Impurities**

▲[Note—Protect solutions containing methyltestosterone from light.]▲ (USP 1-May-2024)

Solution A: Methanol and water (55:45)

Solution B: Methanol

Mobile phase: See *Table 1*.

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	100	0
20	60	40
40	0	100
45	0	100
60	100	0

System suitability solution: ▲0.5 mg/mL of USP Methyltestosterone RS and 0.005 mg/mL of USP Testosterone RS in methanol

Sensitivity solution: 0.25 µg/mL of USP Methyltestosterone RS in methanol

Standard solution: 0.5 µg/mL of USP Methyltestosterone RS in methanol▲ (USP 1-May-2024)

Sample solution: 0.5 mg/mL of Methyltestosterone in methanol

Chromatographic system

(See *Chromatography ⟨621⟩, System Suitability*.)

Mode: LC

Detector: UV 254 nm

Column: 4.6-mm × 25-cm; 5-µm packing L1

▲**Temperatures**

Autosampler: 5°

Column: 30°▲ (USP 1-May-2024)

Flow rate: 1 mL/min

Injection volume: ▲10 µL▲ (USP 1-May-2024)

System suitability

Samples: ▲▲ (USP 1-May-2024) *System suitability solution*, ▲*Sensitivity solution*, and *Standard solution*

[Note—The relative retention times in *Table 2* are provided as information that could aid in peak assignment.]

Table 2

Name	Relative Retention Time
Androstenedione[a]	0.76

Name	Relative Retention Time
Testosterone	0.88
Methyltestosterone	1.0
Epimethyltestosterone[b]	1.31

[a] Androst-4-en-3,17-dione.

[b] 17α-Hydroxy-17-methylandrost-4-en-3-one.

▲ (USP 1-May-2024)

Suitability requirements

▲**Resolution:** NLT 1.5 between testosterone and methyltestosterone, *System suitability solution*▲ (USP 1-May-2024)

Relative standard deviation: NMT ▲5.0%, *Standard solution*▲ (USP 1-May-2024)

Signal-to-noise ratio: NLT ▲10, *Sensitivity solution*▲ (USP 1-May-2024)

Analysis

Samples: ▲*Standard solution* and▲ (USP 1-May-2024) *Sample solution*

▲Calculate the percentage of any specified and unspecified impurity in the portion of Methyltestosterone taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times (1/F) \times 100$$

r_U = peak response of any specified or unspecified impurity from the *Sample solution*

r_S = peak response of Methyltestosterone from the *Standard solution*

C_S = concentration of USP Methyltestosterone RS in the *Standard solution* (mg/mL)

C_U = concentration of Methyltestosterone in the *Sample solution* (mg/mL)

F = relative response factor (see *Table 3*)

▲ (USP 1-May-2024)

Acceptance criteria: ▲See *Table 3*. The reporting threshold is 0.05%.

Table 3

Name	Relative Response Factor	Acceptance Criteria, NMT (%)
Androstenedione	0.98	0.15
Testosterone	1.04	0.10
Epimethyltestosterone	0.97	0.5
Any unspecified impurity	1.0	0.10
Total impurities	—	1.0

▲ (USP 1-May-2024)

SPECIFIC TESTS

- **Optical Rotation** ⟨781S⟩, *Procedures, Specific Rotation*

  Sample solution: 10 mg/mL of Methyltestosterone in alcohol

  Acceptance criteria: +79° to +85°

- **Loss on Drying** ⟨731⟩

  Analysis: Dry at 105° for 4 h.

  Acceptance criteria: NMT 2.0%

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in well-closed, light-resistant containers.
- **USP Reference Standards** (11)
  USP Methyltestosterone RS
  USP Testosterone RS

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
METHYLTESTOSTERONE	Documentary Standards Support	SM52020 Small Molecules 5

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
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