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Determination of Prohibited Minoxidil in Cosmetics - High Performance Liquid Chromatography

化妆品中禁用物质米诺地尔的测定 高效液相色谱法

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1 Scope

This Standard specifies the high-performance liquid chromatography for the determination of prohibited substance of minoxidil in the cosmetics.

This Standard is applicable to the determination of minoxidil in the cosmetics, such as emulsions, creams, lotions and shampoos with hair care and growth functions.

This Standard's detection limit of minoxidil is 5mg/kg; while the quantitative limit is 10mg/kg.

2 Normative References

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this document.

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

3 Principle

The specimen is ultrasonically extracted by the solvent; after centrifuging, determine by high performance liquid chromatography; quantify by external standard method; confirm by liquid chromatography-mass spectrometry.

4 Reagents and Materials

Unless otherwise stated, all the used reagents are analytical reagents; while the used water shall be Class-I water specified in GB/T 6682.

4.1 Methanol: chromatographically pure.

4.2 Trifluoroacetic acid: chromatographically pure.

6.1.2 Lotion and shampoo samples

Take 0.5g (accurate to 0.001g) of specimen; place it into 10mL stoppered plastic centrifuge tube (5.6); add 10mL of 70% methanol aqueous solution (4.5); shake; perform ultrasonic extraction for 20min; after centrifuging for 15min at 5000r/min, filter the supernatant by the 0.45μm microporous membrane; then determine by the high-performance liquid chromatography.

6.2 Determination condition

The HPLC reference conditions are as follows:

a) Chromatographic column: C_{18}, 5μm, 250mm×4.6mm (inner diameter), or the equivalent;

b) Flow rate: 1.0mL/min;

c) Mobile phase: methanol + 0.3% trifluoroacetic acid aqueous solution = 45 + 55 (volume ratio);

d) Detection wavelength: 230nm;

e) Column temperature: 30°C;

f) Sample-injecting volume: 10μL.

6.3 Drawing of the standard curve

Use 70% methanol aqueous solution (4.5) to dilute the minoxidil standard stock solution (4.6) grade by grade into the standard working solutions with concentrations of 0.5mg/L, 1mg/L, 2mg/L, 5mg/L, 10mg/L, and 20mg/L; inject sample and measure according to the low-to-high concentrations of the determination conditions in 6.2; take peak area-concentration as diagram to draw standard working curve.

The high-performance liquid chromatogram of the minoxidil standard substance can refer to Figure A.2 in Appendix A; while the UV absorption spectrum of the minoxidil standard substance can refer to Figure A.3 in Appendix A.

6.4 Determination

Determine the to-be-tested sample as per the determination conditions in 6.2; if the retention time of the chromatographic peak of the detected minoxidil is consistent with the standard substances; in the chromatogram of the sample after subtracting the background, the UV absorption spectrum with such substance is consistent with the standard substance; then it can be preliminarily determined that the minoxidil is present in the sample; use the external standard method to quantify. The minoxidil content shall be within the standard curve; if the linear range is exceeded, then dilute it and analyze.

Appendix B

(Informative)

Confirmation Test

B.1 Liquid chromatography conditions

The reference conditions of liquid chromatography determination are as follows:

a) Chromatographic column: XBridge C_{18}, 3.5μm, 150mm×2.1mm (inner diameter), or the equivalent;

b) Mobile phase: 0.1% formic acid solution + acetonitrile = 10+90 (volume ratio);

c) Flow rate: 0.4mL/min;

d) Column temperature: 40°C;

e) Sample-injecting volume: 5μL.

B.2 Mass spectrometry conditions

The reference conditions of mass spectrometry determination are as follows:

a) Ion source: electrospray ion source;

b) Ionization mode: positive ion mode;

c) Capillary voltage: 3kV;

d) Extraction voltage: 3V;

e) Ion source temperature: 150°C;

f) De-solvent gas: nitrogen;

g) De-solvent gas flow rate: 800L/h;

h) De-solvent gas temperature: 500°C;

i) Data collection method: multiple reaction monitoring (MRM).

B.3 Qualitative determination

When measuring the sample solution, appropriately dilute it; measure the sample solution and standard working solution as per the determination conditions of liquid

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