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Determination of 10 Forbidden Glycol Ethers and Esters in Cosmetics - GC-MS

化妆品中 10 种禁用二元醇醚及其酯类

化合物的测定 气相色谱-质谱法

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1 Scope

This Standard specifies reagents and materials, instruments and equipment, analytical procedures, result calculation, recovery rate and precision, and allowable deviation of gas chromatography-mass spectrometry method of determining 10 forbidden glycol ethers (ethylene glycol dimethyl ether, 2-methoxy ethanol, 2-methoxy propanol, 2-ethoxyethanol, 2-methoxy propyl acetate, 2-methoxyethyl acetate, 2-ethoxy ethyl acetate, dimethoxy diglycol, 2-(2-methoxye-thoxy) ethanol, triethylene glycol dimethyl ether) and their ester compounds in cosmetics.

This Standard is applicable to the determination of the content of 10 forbidden glycol ethers and their ester compounds in cosmetics.

Please refer to Table A.1 in Appendix A for the detection limit and quantitation limit of the method in this Standard.

2 Principle

Sample goes through ultrasonic extraction with ethyl acetate-methanol mixed solvent. Adopt gas chromatograph-mass spectrometer for determination; quantify through the external standard method.

3 Reagents and Materials

Unless it is otherwise indicated, all reagents being adopted shall be analytical pure.

3.1 Ethyl acetate: chromatographic purity.

3.2 Methanol: chromatographic purity.

3.3 Anhydrous sodium sulfate.

3.4 20% ethyl acetate methanol solution (V/V, volume ratio): measure-take 200 mL of ethyl acetate (3.1), then, mix it up with 800 mL of methanol (3.2).

3.5 Standard samples/substances of 10 forbidden glycol ethers and esters: purity shall be not less than 98%. Please refer to Table B.1 in Appendix B for the Chinese name, English name, CAS No., molecular formula, relative molecular mass and chemical

same as the procedures of non-waxy based sample treatment.

5.2 Determination

Since the determination result depends on the instrument being adopted, it is impossible to provide general parameters of gas chromatography-mass spectrometry analysis. The set parameters shall guarantee that the tested components and the other components can be effectively separated in chromatographic determination. The reference conditions of gas chromatography-mass spectrometry are as follows:

a) Chromatographic column: VF-WAX column, 60 m x 0.25 mm (i.d.) x 0.25 μm, or other equivalents;

b) Inlet temperature: 220 °C;

c) Transmission line temperature: 280 °C;

d) Inlet mode: split injection, split ratio: 5:1;

e) Carrier gas: helium, purity ≥ 99.999%; mode of control: constant flow; flow rate: 1.0 mL/min;

f) Column temperature: initial temperature: 50 °C, maintain for 7 min; rise to 140 °C at 5 °C/min, maintain for 1 min; rise to 240 °C at 20 °C/min, maintain for 2 min. Post-treatment temperature: 245 °C, duration: 3 min.

g) Inlet volume: 1 μL.

h) Ionization source: EI source;

i) Ionization energy: 70 eV;

j) Quadrupole temperature: 150 °C;

k) Ionization source temperature: 230 °C;

l) Solvent delay time: 7.8 min;

m) Mode of monitoring: select ion scanning mode (SIM); please refer to Table C.1 in Appendix C for monitoring ions.

5.3 Blank Test

Other than sample-weighing, comply with procedure 5.1 and procedure 5.2.

5.4 Qualitative Confirmation

Under the instrument conditions in 5.2, the selected ion chromatographic peak of the tested sample and the standard solution emerges in the same retention time, and the

V---the constant volume of the sample, expressed in (mL);

m---the sample mass represented by the ultimate test solution, expressed in (g).

The calculation result shall retain 3 significant figures.

7 Recovery Rate and Precision

Non-waxy based sample shall be within the fortified concentration range of 2.0 mg/kg ~ 30 mg/kg; within the recovery rate of 80% ~ 110%; its relative standard deviation shall be less than 10%.

Waxy-based sample shall be within the fortified concentration range of 3.0 mg/kg ~ 60 mg/kg; within the recovery rate of 80% ~ 110%; its relative standard deviation shall be less than 10%.

8 Allowable Deviation

The absolute difference of two independent determination results obtained under repeatability conditions shall not exceed 10% of the arithmetic mean value.

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