GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 71.100.70

Y 42

GB/T 35829-2018

Determination of Four Naphthalenediols in Cosmetics - High Performance Liquid Chromatography

化妆品中 4 种萘二酚的测定 高效液相色谱法

Issued on: February 06, 2018 **Implemented on: September 01, 2018**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine;

Standardization Administration of PRC.

Table of Contents

Determination of Four Naphthalenediols in Cosmetics - High Performance Liquid Chromatography

1 Scope

This Standard specifies high performance liquid chromatography for the determination of 1,7-naphthalenediol, 2,3-naphthalenediol, 1,5-naphthalenediol and 2,7-naphthalenediol in cosmetics.

This Standard is applicable to the determination of 1,7-naphthalenediol, 2,3-naphthalenediol, 1,5-naphthalenediol and 2,7-naphthalenediol in non-wax-based cream, lotion, gel, water and powder cosmetics.

The method detection limit of this Standard: the method detection limit of four naphthalenediols is 2.5mg/kg.

The method quantification limit of this Standard: the method quantification limit of four naphthalenediols is 7.5mg/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 Principles

The samples of cream, lotion, gel and water cosmetics shall be directly extracted by 95% ethanol; the samples of powder cosmetics shall be extracted by the mixture of 95% ethanol and 0.1% acetic acid solutions; the extracting solution shall be determined by high-performance liquid chromatography after the filtering and centrifuging. According to the retention time and spectrogram qualitative, use the external standard quantitative method; if necessary, use liquid chromatography-mass spectrometry/mass spectrometry to verify.

6 Analytical Procedures

6.1 Specimen processing

6.1.1 Cream, lotion and water specimens

Take 0.5g (accurate to 0.001g) of specimen; place it into 10mL stoppered colorimetric tube; add 4mL of 95% ethanol (4.3); shake and mix on the vortex oscillator (5.3); then perform ultrasonic extraction for 15min; cool off to the room temperature; use 95% ethanol (4.3) to make constant volume to 5mL. Take 2mL of solution into the centrifuge tube; centrifuge for 2min at 10000r/min; filter the supernatant through a filter membrane (4.8) then determined by the high-performance liquid chromatography (5.1).

6.1.2 Powder specimen

Take 0.5g (accurate to 0.001g) of specimen; place it into 10mL stoppered colorimetric tube; add 2mL of 95% ethanol (4.3); shake and mix on the vortex oscillator (5.3); then add 2mL of 0.1% acetic acid solution (4.4); perform ultrasonic extraction for 15min; cool off to the room temperature; use 95% ethanol (4.3) to make constant volume to 5mL. Take 2mL of solution into the centrifuge tube; centrifuge for 2min at 10000r/min; filter the supernatant through a filter membrane (4.8) then determined by the high-performance liquid chromatography (5.1).

6.2 Determination

6.2.1 Reference operating conditions of high-performance liquid chromatograph

The reference operating conditions of high-performance liquid chromatograph are as follows:

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

b) Mobile phase: methanol (4.1) and 0.1% acetic acid solution (4.4), high-performance liquid chromatograph gradient elution conditions are shown in Table 1;

c) Flow rate: 1.0mL/min;

d) Column temperature: 30°C;

e) Wavelength: 230nm;

f) Sampling-injecting volume: 20μL.

$$X_i = \frac{(c_i - c_0) \times V \times 1\,000}{m \times 1\,000} \times f$$

Where:

X_i – the content of the test composition in the specimen; in mg/kg;

c_i – the mass concentration of the test composition in the specimen solution obtained from the standard curve; in μg/mL;

c_0 – the mass concentration of the test composition in the blank sample obtained from the standard curve; in μg/mL;

V – the constant volume of the specimen solution, in mL;

m – specimen mass, in g;

f – dilution factor.

The results shall be retained two significant figures.

8 Recovery Rate and Precision

In the range of add concentration of 7.5mg/kg ~75mg/kg, the recovery rate is 80%~110%; the relative standard deviation is less than 10%.

9 Allowable Deviation

The absolute difference between two independent determinations obtained under repeatability conditions shall not exceed 10% of the arithmetic mean.

Appendix C

(Informative)

Confirmation Test

C.1 Liquid chromatography reference conditions

The liquid chromatography reference conditions are as follows:

a) Chromatographic column: Luna C_{18}(2), 150×2.0mm (inner diameter), 3μm; or the equivalent;

b) Mobile phase: A – methanol; B – water; gradient elution: 0.0min~10.0min, 25%~65%A; 10.1min~30.0min, 65%A.

c) Flow rate: 150μL/min;

d) Column temperature: 25°C;

e) Sample-injecting volume: 10μL.

C.2 Mass spectrometry reference conditions

The mass spectrometry reference conditions are as follows:

a) Ion source: electrospray ion source;

b) Ionization mode: negative ion mode;

c) Ionization voltage: -4500V;

d) Atomizing gas pressure: 0.345MPa (50psi);

e) Air curtain gas pressure: 0.241MPa (35psi);

f) Auxiliary heating gas pressure: 0.483MPa (70psi);

g) Auxiliary gas temperature: 500°C;

h) Detection mode: Multiple Reaction Monitoring (MRM) mode. The mass spectrometric analysis parameters of four naphthalenediols can refer to Table C.1; the multiple reaction monitoring mass chromatograms of four naphthalenediols can refer to Figure C.1.

C.3 Qualitative determination

This is an excerpt of the PDF (Some pages are marked off intentionally)

------ The End ------