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NATIONAL STANDARD OF THE
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GB/T 42425-2023

**Determination of capryloyl salicylic acid, phenylethyl
resorcinol, ferulic acid in cosmetics - High performance
liquid chromatography**

化妆品中功效组分辛酰水杨酸、苯乙基间苯二酚、阿魏酸的测定
高效液相色谱法

Issued on: March 17, 2023

Implemented on: October 1, 2023

**Issued by: State Administration for Market Regulation;
Standardization Administration of PRC.**

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Determination of capryloyl salicylic acid, phenylethyl resorcinol, ferulic acid in cosmetics - High performance liquid chromatography

WARNING -- The personnel using this document shall have practical experience in formal laboratory work. This document does not indicate all possible safety issues. The user is responsible for taking appropriate safety and health measures and ensuring compliance with the conditions stipulated by relevant national regulations.

1 Scope

This document describes the high-performance liquid chromatography method for the determination of capryloyl salicylic acid, phenylethyl resorcinol, and ferulic acid in cosmetics, including principles, reagents and materials, instruments and equipment, test steps, test data processing, recovery rate, precision, etc.

This document is applicable to the determination of capryloyl salicylic acid, phenylethyl resorcinol, and ferulic acid in water, lotion, and cream cosmetics.

In this document, the method detection limit and quantification limit of capryloyl salicylic acid, phenylethyl resorcinol, and ferulic acid are 3.3 mg/kg and 10 mg/kg, respectively.

2 Normative references

The following documents contain the provisions which, through normative reference in this document, constitute the essential provisions of this document. For the dated referenced documents, only the versions with the indicated dates are applicable to this document; for the undated referenced documents, only the latest version (including all the amendments) is applicable to this document.

GB/T 6682 Water for analytical laboratory use - Specification and test methods

3 Terms and definitions

There are no terms and definitions that need to be defined in this document.

4 Principles

The sample is vortex-oscillated with methanol solution, treated through ultrasonic extraction, centrifugal precipitation, separation and filtration, separated by reversed-phase high-performance liquid chromatography, detected by a diode array detector, and quantified by external standard method.

5 Reagents and materials

Unless otherwise specified, only analytically pure reagents are used.

5.1 Water: Grade 1 specified in GB/T 6682.

5.2 Acetonitrile (chromatographically pure).

5.3 Methanol.

5.4 Capryloyl salicylic acid, phenylethyl resorcinol, and ferulic acid: The content is not less than 95%. See Table A.1 in Appendix A for the English name, INCI name (International Name of Cosmetic Ingredients), CAS number, molecular formula, relative molecular mass, and chemical structural formula.

5.5 Sea sand, chemically pure.

5.6 Sodium decanesulfonate.

5.7 Phosphoric acid.

5.8 Standard stock solution of capryloyl salicylic acid, phenylethyl resorcinol, and ferulic acid: Accurately weigh 25.0 mg of each of capryloyl salicylic acid, phenylethyl resorcinol, and ferulic acid (5.4) in a 25 mL volumetric flask, and dilute to the mark with methanol (5.3). A standard stock solution with a mass concentration of 1000 µg/mL is obtained; it is stored in a -18 °C refrigerator, and the validity period is 6 months.

5.9 Polytetrafluoroethylene (PTFE) filter membrane: The pore size is 0.22 µm.

6 Instruments and equipment

6.1 High-performance liquid chromatograph (diode array detector).

6.2 Analytical balance: The sense quantity is 0.1 mg and 0.001 g.

6.3 Volumetric flask: 10 mL, 25 mL, and 100 mL.

6.4 Graduated centrifuge test tube with a stopper: 10 mL.

6.5 Ultrasonic oscillator: The power is not less than 180 W.

6.6 Centrifuge: The rotation speed is not less than 10000 r/min.

6.7 Vortex oscillator.

6.8 Pipette or pipettor: 1 mL, 5 mL, and 10 mL.

7 Test steps

7.1 Sample treatment

For water cosmetics, take 1 g of the sample (accurate to 0.001 g) into a graduated centrifuge tube with a stopper, add 7 mL of methanol (5.3), vortex-oscillate for 1 minute, extract with ultrasonic for 20 minutes, and cool to room temperature; make up the volume to 10 mL with methanol (5.3), centrifuge at 10000 r/min for 10 min, and filter the supernatant through a 0.22 μm filter membrane (5.9) for later testing.

For facial masks (the mask liquid taken from facial masks), lotions, and cream cosmetics, take 1 g of the sample (accurate to 0.001 g) into a graduated centrifuge tube with a stopper, add methanol (5.3) to make up the volume to 10 mL, vortex-oscillate for 1 minute, and conduct ultrasonic extraction for 20 min; after cooling to room temperature, add 0.5 g~1.0 g of sea sand (5.5), vortex for 1 min, centrifuge at 10000 r/min for 10 min, and filter the supernatant through a 0.22 μm filter membrane (5.9) for later testing.

Do two experiments in parallel.

7.2 Determination conditions

The reference conditions for high-performance liquid chromatography determination are as follows:

- a) Chromatographic column specifications: C₁₈ chromatographic column, 250 mm×4.6 mm (particle size of 5 μm); or, a chromatographic column with equivalent performance;
- b) Mobile phase: 5 mmol/L sodium decanesulfonate solution (containing 0.1% phosphoric acid) (A): acetonitrile (B), and the gradient elution procedure is shown in Table 1;
- c) Column temperature: 35 °C;
- d) Mobile phase flow rate: 1.0 mL/min;

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