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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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

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Foreword

This document was issued on 17 March 2023 by the State Administration for Market Regulation; Standardization Administration of the PRC and takes effect on 1 October 2023.

It is a GB/T standard: recommended rather than compulsory, but it is the text a Chinese reviewer applies when assessing a submission.

1 Scope

GB/T 42425-2023 measures three declared actives in finished cosmetics - capryloyl salicylic acid, phenylethyl resorcinol and ferulic acid - by high performance liquid chromatography. The principle clause fixes the route: the sample is vortex-mixed with methanol solution, extracted under ultrasound, centrifuged, separated and filtered, then run on reversed-phase liquid chromatography with a diode array detector. Reagents and materials - grade 1 water to GB/T 6682, chromatographically pure acetonitrile - and the instruments and equipment, from the chromatograph and its detector down to the balance and the volumetric flasks, are listed so the chemistry is not left to each laboratory's habits. Test steps then take the sample through preparation and measurement, and result calculation, recovery rate and precision close the method. These three ingredients are what a label sells the product on: a keratolytic, a skin-lightening agent and an antioxidant, each of them the subject of a claim and of a concentration a regulator can check. Without one agreed extraction and separation, a supplier's certificate and a market surveillance result can disagree far enough to condemn a compliant batch or to clear a non-compliant one.

Cosmetic manufacturers, ingredient suppliers, contract laboratories and inspection authorities are the readers.

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 ug/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 um.

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 um 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 um 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 mmx4.6 mm (particle size of 5 um); 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 degrees C;

d) Mobile phase flow rate: 1.0 mL/min;

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