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**Determination of 10 whitening and freckle-removing
components in cosmetics - High performance liquid
chromatography**

化妆品中 10 种美白祛斑剂的测定
高效液相色谱法

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Determination of 10 whitening and freckle-removing components in cosmetics - High performance liquid chromatography

1 Scope

This Standard specifies reagents and materials, instruments and equipment, analytical procedures, calculation of results, recovery and precision, allowable difference, etc. for high performance liquid chromatography of 10 whitening and freckle-removing components (magnesium ascorbyl phosphate, ascorbyl glucoside, β -arbutin, kojic acid, nicotinamide, 3-O-ethylascorbic acid, potassium methoxysalicylate, raspberry glucoside, dipotassium glycyrrhizinate, 4-butyl resorcinol) in cosmetics.

This Standard applies to the determination of whitening and freckle-removing components in water-based, lotion, cream, gel, wax-based, powder-based cosmetics.

For the detection limits and quantification limits of this Standard method, see Table A.1 in Annex A.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

3 Method summary

The sample takes potassium dihydrogen phosphate solution as extraction solvent. Perform direct ultrasonic extraction of water-based samples and oil-in-water samples. Water-in-oil samples, wax-based, and powder-based samples are dissolved and dispersed in dichloromethane, and then extracted with ultrasonic solvent. Perform centrifugation, filtration through a 0.22 μ m water filter. Inject solution into a liquid chromatograph equipped with a diode array detector

20.0mg/L, 50.0mg/L, 100.0mg/L.

5 Instruments and equipment

5.1 High performance liquid chromatograph equipped with diode array detector (DAD).

5.2 Centrifuge: >9500r/min.

5.3 Analytical balance: resolutions are 0.0001g and 0.001g.

5.4 PH meter.

5.5 Vortex mixer.

5.6 Ultrasonic cleaner.

5.7 Water microporous membrane: 0.45μm.

6 Analytical procedures

6.1 Sample processing

6.1.1 Preparation of water-based, oil-in-water samples

Weigh 0.5g of (to the nearest of 0.001g) sample in a 50mL colorimetric tube. Add 30mL of potassium dihydrogen phosphate solution (4.4). Conduct vortex oscillation until the sample is completely dispersed. Perform ultrasonic extraction for 20min. Transfer to a 50mL volumetric flask. Use potassium dihydrogen phosphate solution (4.4) to set volume to the scale. After mixing evenly, take an appropriate amount of solution into the centrifuge tube. Centrifuge at 9500r/min for 5min. Take the upper aqueous phase solution after centrifugation. Filter through a 0.45μm water filter, the filtrate is to be determined.

6.1.2 Preparation of water-in-oil, wax-based, powder-like samples

Weigh approximately 0.5g of (to the nearest of 0.001g) sample in a 50mL stoppered centrifuge tube. Add 2.5mL of dichloromethane (4.2). Vortex until the sample is completely dispersed (if a small amount of sample is not dispersed, it can be sonicated to complete dispersion). Add 20mL of potassium dihydrogen phosphate solution (4.4). Perform ultrasonic extraction for 20min, vortexing for 1min, centrifugation at 9500r/min for 5min. Remove the supernatant into a 50mL volumetric flask. Add 20mL of potassium dihydrogen phosphate solution (4.4). Vortex for 1min, centrifuge at 9500r/min for 5min, and combine the supernatant into a 50mL volumetric flask. Use potassium dihydrogen

Take the mixed standard working solution (4.8) and determine according to the chromatographic conditions (6.2.1). Taking the peak area (Y) as the ordinate and the standard solution mass concentration (X, mg/L) as the abscissa, draw a standard working curve. For the liquid chromatogram of the standard solution of whitening and freckle-removing components, see Figure C.1 in Annex C.

6.2.3 Specimen determination

The sample solution to be tested (6.1) is sequentially injected into the liquid chromatograph. Determine according to chromatographic conditions (6.2.1). Record the retention time and peak area of the chromatographic peak. Calculate the content of the target compound in the sample solution according to the standard curve. The response of the measured object in the sample solution shall be within the linear range of the standard curve. Specimen exceeding the linear range may be diluted with potassium dihydrogen phosphate solution (4.4) and then determined.

NOTE: In order to ensure the accuracy of specimen quantification, try to complete the test process within 12h.

6.2.4 Qualitative confirmation

Perform qualitative determination of sample by liquid chromatograph. When the sample is being determined, if the retention time of the peak of the tested whitening and freckle-removing component is consistent with the standard, and the chromatogram of the sample after subtracting the background, the UV absorption of the substance is consistent with the UV absorption of the standard, it can be preliminarily determined that the sample contains the measured whitening and freckle-removing component. If necessary, other methods are needed to confirm.

6.3 Blank test

Except for not weighing samples, they are carried out according to the above measurement conditions and procedures.

6.4 Parallel test

According to the above steps, perform parallel test determination of the same sample.

7 Calculation of result

The content of whitening and freckle-removing component in the sample is calculated by the following formula:

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