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Determination of Undecylenic Acid and Zinc Undecylenate in Cosmetics - Gas Chromatography

化妆品中十一烯酸及其锌盐的测定 气相色谱法

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Determination of Undecylenic Acid and Zinc Undecylenate in Cosmetics - Gas Chromatography

1 Scope

This Standard specifies the gas chromatographic method for the determination of undecylenic acid and zinc undecylenate in cosmetics (creams, emulsions, lotions) and hand sanitizers.

This Standard applies to the determination of undecylenic acid and zinc undecylenate in cosmetics (creams, emulsions, lotions) and hand sanitizers.

The detection-limit of this method for undecylenic acid and zinc undecylenate is 12 mg/kg (0.001 2%), and the quantitation-limit is 40 mg/kg (0.004%) (both in terms of acid).

2 Normative references

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

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

3 Principle

After the undecylenic acid and zinc undecylenate are methylated by the sulfuric acid-methanol solution, use n-hexane to extract the produced methyl undecylenate; filter the n-hexane layer through a 0.45 μm filter; then, inject it into the gas chromatograph for detection; use the external standard method to quantify.

4 Reagents and materials

Unless otherwise specified, all the reagents are analytical reagents, and the water is grade-1 water which is specified by GB/T 6682.

4.1 Methanol: chromatographic pure.

(4.2); vortex and mix well; add 2 mL of saturated sodium chloride solution (4.8); vortex and mix well; let stand for layering; add 2 mL of distilled water to wash the organic layer; let stand; take the upper organic layer for filtering and injection.

6.2 Sample treatment

Accurately weigh 0.25 g of sample; add 5 mL of 5% sulfuric acid-methanol solution (4.7); perform ultrasonic extraction for 10 min. The methyl esterification steps are the same as 6.1.

6.3 Determination

6.3.1 Chromatographic conditions

The chromatographic conditions are shown as below:

a) chromatographic column: HP-5 capillary column (30.0 m × 0.25 mm × 0.25 μm) or a column of equivalent polarity;

b) heating procedure: keep the initial temperature of 100°C for 2 min; rise the temperature to 240°C at 15°C/min; then, keep it for 5 min;

c) temperature of the injector: 250°C; temperature of the detector: 260°C;

d) carrier gas flow velocity: 1 mL/min.

e) injection volume: 1 μL;

f) split ratio: 30:1.

6.3.2 Drawing of the standard working curve

Inject the undecylenic acid standard working solution (6.1) into the gas chromatograph; detect it according to the chromatographic conditions of 6.3.1; respectively take the chromatographic peak area of the undecylenic acid methyl ester as the ordinate, and the total content of undecylenic acid in the system before methyl esterification as the abscissa to draw the standard working curve of undecylenic acid. See Figure B.1 in Appendix B for the gas chromatogram of methyl esterification of undecylenic acid standard solution.

6.3.3 Sample determination

Inject the sample solution (6.2) into the gas chromatograph; measure according to the chromatographic conditions of 6.3.1. The peak area of the chromatographic peak can be used to determine the total undecylenic acid content from the standard curve; the response value of the measured substance in the sample solution shall be within the linear range of the standard curve. When the content of the measured substance is higher than the upper

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