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Method of Test for α-Hydroxy Acids in Cosmetics

1. Scope

This method is applicable to the determination of glycolic acid, dl-malic acid, lactic acid, and citric acid in cosmetics.

2. Method

After extraction, analytes are determined by high performance liquid chromatography (HPLC).

2.1. Equipment

2.1.1. High performance liquid chromatograph.

2.1.1.1. Detector: photodiode array detector.

2.1.1.2. Column: Capcell PAK C18 UG120, 5 μm, 4.6 mm i.d. × 25 cm, or an equivalent product.

2.1.2. Ultrasonicator.

2.2. Chemicals

Methanol, HPLC grade;
Phosphoric acid (85%), reagent grade;
Ammonia solution (25%), reagent grade;
Deionized water, resistivity ≥ 18 MΩ • cm (at 25°C);
Glycolic acid, dl-malic acid (99%, contain <1% furamic acid impurity), lactic acid, and citric acid, reference standards.

2.3. Apparatus

2.3.1. Volumetric flask: 10 mL and 20 mL.

2.3.2. Membrane filter: 0.45 μm, Nylon.

2.4. Mobile phase

Dilute 20 mL of phosphoric acid with deionized water to 1000 mL, adjust pH to 2.0 with ammonia solution, and filter with a membrane filter.

2.5. Standard solution preparation

Transfer about 10 mg of glycolic acid, dl-malic acid, lactic acid, and citric acid reference standards accurately weighed into each 10-mL volumetric flask, dissolve and dilute with deionized water to volume as the standard stock solutions. Store in the refrigerator. When to use, mix appropriate amount of each standard stock solution, and dilute with deionized water to 25-500 μg/mL as the standard solutions.

2.6. Sample solution preparation

Transfer about 1 g of the well-mixed sample accurately weighed into a 20-

mL volumetric flask, add 15 mL of deionized water, and ultrasonicate for 30 mins. Dilute to volume with deionized water, and filter with a membrane filter. Take the filtrate as the sample solution.

2.7. Identification and quantification

Accurately inject 20 μL of the sample solution and the standard solutions into HPLC respectively, and operate according to the following conditions. Identify each α-Hydroxy acid based on the retention time and the UV absorption spectrum. Calculate the amount of each α-Hydroxy acid in the sample by the following formula:

$$\text{The amount of each α-Hydroxy acid in the sample (\%)} = \frac{C \times V}{M} \times 10^{-4}$$

where,

C: the concentration of each α-Hydroxy acid in the sample solution calculated by the standard curve (μg/mL)

V: the final make-up volume of sample (mL)

M: the weight of the sample (g)

HPLC operating conditions (note):

Photodiode array detector: the quantitative wavelength 210 nm.

Column: Capcell PAK C18 UG120, 5 μm, 4.6 mm i.d. × 25 cm.

Mobile phase: as section 2.4.

Flow rate: 0.5 mL/min.

Injection volume: 20 μL.

Note: All the parameters can be adjusted depending on the instruments used if the above conditions are not applicable.

Remark

1. Limits of quantification (LOQs) are 0.05% for glycolic acid, dl-malic acid, lactic acid, and citric acid.
2. Further validation should be performed when interference compounds appear in samples.

Reference

1. Huang, W. S., Lin, C. C., Huang, M. C. and Wen, K. C. 2002. Determination of α-hydroxy acids in cosmetics. J. Food Drug Anal. 10: 95-100.
2. Cherchi, A., Spanedda, L., Tuberoso, C. and Cabras, P. 1994. Solid-phase extraction and high-performance liquid chromatographic determination of organic acid in honey. J. Chromatogr. A 669: 59-64.

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Reference chromatogram

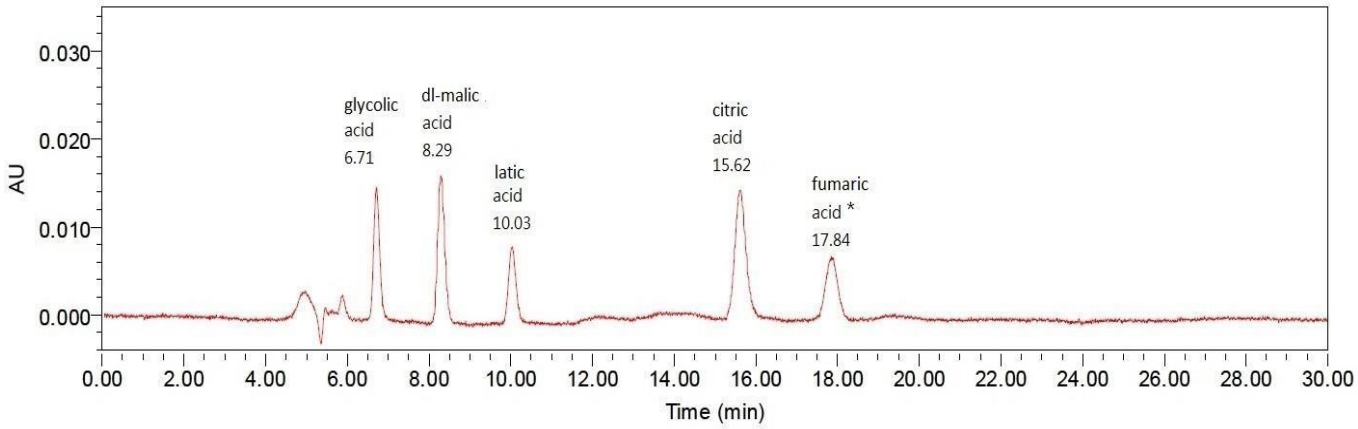

Figure. HPLC chromatogram of α-Hydroxy acid reference standards

* Fumaric acid is the impurity of dl-malic acid.