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**Determination of restricted preservatives in
toothpaste - High performance liquid chromatography
method**

牙膏中限用防腐剂的测定 高效液相色谱法

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Determination of restricted preservatives in toothpaste - High performance liquid chromatography method

1 Scope

This Standard specifies high performance liquid chromatography (HPLC) method to determine the content of restricted preservatives - sorbic acid and benzoic acid - in toothpaste.

This Standard is applicable to the determination of the content of restricted preservatives - sorbic acid and benzoic acid - in toothpaste.

The method detection limit of this Standard is 0.5mg/kg.

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 6682-2008, *Water for analytical laboratory use - Specification and test methods*

3 Principle

Use pure water to extract the sample. The supernatant is filtered through a 0.45µm filter membrane. Use high performance liquid chromatography-ultraviolet detection method to determine. Use retention time to characterize. Use external standard peak area method to quantify.

4 Reagents and standard materials

Unless otherwise specified, all reagents used are analytically-pure AND water is grade one water specified in GB/T 6682-2008.

4.1 Methanol: chromatographically pure.

value to 7. After shaking for 15min, add pure water to set volume to 25mL. Shake well. Let it still. Transfer the supernatant to the centrifuge tube. Centrifuge at 4000r/min for 10min. Take the supernatant and pass it through the 0.45 μ m microporous membrane. Inject a sample to test.

7 Determination by high performance liquid chromatography

7.1 Reference conditions of liquid chromatography

The reference conditions of liquid chromatography are as follows:

- a) Chromatographic column: C₁₈ stainless steel column (250mm×4.6mm×5 μ m).
- b) Detection wavelength: 230nm.
- c) Mobile phase: methanol/ammonium acetate aqueous solution (0.02mol/L) (5:95, volume ratio).
- d) Flow rate: 1.0mL/min.
- e) Column temperature: 30°C.
- f) Injection volume: 10 μ L.

7.2 Preparation of standard working solution

Use mobile phase solution to gradually dilute the 100mg/L mixed standard solution (4.7). Prepare into 50mg/L, 20mg/L, 10mg/L, 5mg/L standard working solutions. Filter by 0.45 μ m filter membrane for future use. Under the set chromatographic conditions, respectively take 10 μ L for analysis. Take the mass concentration of the standard series of solutions as the abscissa (mg/L), the peak area of the reference substance as the ordinate to draw the calibration curve (linear correlation coefficient $R \geq 0.9990$). Refer to Figure A.1 in Annex A for the liquid chromatogram of the mixed benzoic acid and sorbic acid standard sample.

7.3 Determination steps

After the sample is processed according to the steps in Chapter 6, perform on-board testing according to the instrument conditions of 7.1. The response values of benzoic acid and sorbic acid in the test sample solution shall be within the linear range of the standard curve. If it exceeds the linear range, it shall be diluted before injection for determination.

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