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Replacing GB 8372-2008

Toothpaste

牙膏

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Standardization Administration of the PRC.

Table of Contents

Toothpaste

1 Scope

This Standard specifies the terms and definitions, requirements, test methods, inspection rules, marking, transportation and storage of toothpaste.

This Standard applies to toothpaste for cleansing and care of the mouth.

2 Normative references

The following documents are indispensable for the application of this document. For the dated references, only the editions with the dates indicated are applicable to this document. For the undated references, the latest edition (including all the amendments) are applicable to this document.

GB/T 191 Packaging - Pictorial Marking for Handling of Goods

GB/T 601 Chemical Reagent - Preparations of Standard Volumetric Solutions

GB/T 602 Chemical Reagent - Preparations of Standard Solutions for Impurity

GB/T 603 Chemical Reagent - Preparations of Reagent Solutions for Use in Test Methods

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

GB 7917.2 Standard methods of hygienic test for cosmetics - Arsenic

GB 22115 General requirements on raw materials of toothpastes

GB 29337 General labeling for oral care and cleansing products

JJF 1070 Rules of Metrological Testing for Net Quantity of Products in Prepackages with Fixed Content

Measures for the Metrological Supervision and Administration of Products in Prepackages with Fixed Content (No. 75 Decree [2005] by the General Administration of Quality Supervision, Inspection and Quarantine)

Safety and Technical Standards for Cosmetics (China Food and Drug

4.6 Packaging appearance requirements

4.6.1 Hose or other packaging

The cap and the hose mouth are tightly matched. There shall be no damage to the hose body and no paste leakage.

4.6.2 Box (for toothpaste packaged in small box)

The box shall be free from damage.

5 Test methods

The reagents and water used in this Standard, unless otherwise specified, are analytically pure reagents and water in accordance with GB/T 6682.

In this Standard, standard solutions for titrimetric analysis, standard solutions for impurity determination, and preparations and products used in test methods, unless otherwise specified, are prepared according to the provisions of GB/T 601, GB/T 602, and GB/T 603.

For indicators whose final test result is calculated by the weighing method, use the sampling method of first squeezing 20 mm of paste out and discarding and then squeezing again and weighing.

5.1 Microbiological indicators

It shall be in accordance with Clause 5 Microbiological inspection methods of “Safety and Technical Standards for Cosmetics”.

5.2 Lead (Pb) content

5.2.1 General

This method is an arbitration inspection method. Non-arbitration inspection may be carried out in accordance with the provisions of Appendix A.

5.2.2 Toothpaste with calcium carbonate or calcium hydrogen phosphate as friction agent

5.2.2.1 Reagents

5.2.2.1.1 Nitric acid.

5.2.2.1.2 Nitric acid solution: 5 mol/L.

5.2.2.1.3 Nitric acid solution: 0.2 mol/L.

shake; HEAT on small fire until hydrogen peroxide completely decomposes. If reddish brown nitrogen dioxide fumes are generated, immediately add 2 mL of 10% ammonium sulfamate solution and heat until the solution boils slightly; quickly add water to 50 mL, to let it cool rapidly; ADD 3 mL of aqueous ammonia; COOL to room temperature and then transfer the solution to a 100 mL beaker; USE 10 mL of water to wash the conical flask twice. USE aqueous ammonia and 1% ammonium hydroxide solution to adjust the pH to 1.1~1.2. At this point, a small amount of turbidity will appear in the solution. It is filtered into a 125 mL separatory funnel. USE 5 mL of water to wash the beaker and funnel. ADD 1 mL of 2% ammonium tetrahydropyrrole dithiocarbamate solution to the solution and shake well; PLACE it for about 3 min; ADD 10 mL of trichloromethane and shake for 2 min. After layering, transfer the trichloromethane layer to another separatory funnel; then use 10 mL of trichloromethane to repeatedly extract; COMBINE the extracts. ADD 10.0 mL of Hg^{2+} solution and shake for 2 min; after layering, take the upper aqueous phase for flame atomic absorption determination. At the same time, use 0.01 mol/L nitric acid solution as blank; determine the absorbance of lead standard series 1 μg/mL, 3 μg/mL, 5 μg/mL. USE the lead concentration as the abscissa and the lead absorbance as the ordinate, to draw a standard curve.

5.2.3 Toothpaste with silica or aluminum hydroxide as friction agent

5.2.3.1 Reagents

Sulfuric acid.

Other reagents are the same as 5.2.2.1.

5.2.3.2 Apparatus

Same as 5.2.2.2.

5.2.3.3 Preparation and determination of sample

5.2.3.3.1 Silica toothpaste

Randomly take 1 sample toothpaste, from which weigh 2 g of toothpaste, accurate to 0.01 g; PLACE it in a 250 mL conical flask; ADD 5 mL of water and 5 mL of nitric acid; USE a small fire to heat, until the paste dissolves. Slightly cool, add 1.5 mL of hydrogen peroxide solution and shake; USE a small fire to heat, until reddish brown nitrogen dioxide gas generates. Immediately add 2 mL of 10% ammonium sulfamate solution; REMOVE when slightly hot; ADD 20 mL of water; COOL to room temperature; USE two layers of filter paper to suction-filter. USE 15 mL of water to wash the conical flask and the inner wall of Buchner funnel and the precipitate several times; TRANSFER the suction-filtrate to a 100 mL beaker; USE 10 mL of water to wash the filter bottle twice; adjust the

5.5.2 Determination procedure

Randomly take 1 sample toothpaste, from which weigh 5 g of toothpaste, accurate to 0.01 g. PLACE it in a 50 mL beaker; ADD 20 mL of pre-boiled and cooled distilled water and stir well; at 20 ° C, use a pH meter to measure; within 10 min, read the indicated value.

5.5.3 Result representation

The absolute difference of the parallel measured values is not more than 0.1pH unit. The arithmetic mean is taken as the measurement result.

5.6 Stability

5.6.1 Apparatus

5.6.1.1 Refrigerator: Accuracy is ±1 °C.

5.6.1.2 Electrothermal constant-temperature incubator: Accuracy is ±1 °C.

5.6.2 Determination procedure

TAKE 2 sample toothpaste. 1 sample is stored at room temperature. The other sample is placed in a refrigerator at -8 °C±1 °C. After 8 h, it is taken out; then placed in a constant-temperature incubator at 45 °C±1 °C and taken out after 8 h. Restore to room temperature; OPEN the lid; the paste shall not overflow the hose mouth. INVERT the toothpaste hose. Within 10 s, no liquid shall drip out from the hose mouth. After the paste is squeezed out, it is compared with the sample stored at room temperature. The fragrance and color shall be normal.

5.7 Hard particles

5.7.1 Apparatus

5.7.1.1 Hard particle tester: 1.

5.7.1.2 Slide: 75 mm×25 mm.

5.7.2 Reagents

Nitric acid: 1+1.

5.7.3 Determination procedure

TAKE 1 sample toothpaste, from which weigh 5 g of toothpaste onto a scratch-free slide; PUT the slide into the fixed slot of the tester; PRESS a friction copper block on; START the switch, to make the copper block reciprocally rub 100 times. After that, stop the friction; TAKE the slide out; USE water or hot nitric

respectively. ADD 5 mL of citrate buffer respectively; USE deionized water to dilute to the mark; then transfer to 50 mL plastic beaker one by one. Under magnetic stirring, measure the potential value E. RECORD and make an E-log c (c is concentration) standard curve.

5.8.5 Determination of soluble fluorine

Accurately pipette 2.0 mL of the supernatant prepared in 5.8.3 and transfer to a 5 mL microcentrifuge tube. ADD 0.7 mL of 4 mol/L hydrochloric acid; COVER the centrifuge tube; PUT in a 50 °C water bath for 10 min; TRANSFER to a 50 mL plastic volumetric flask; ADD 0.7 mL of 4 mol/L sodium hydroxide to neutralize and add 5 mL of citrate buffer. USE deionized water to dilute to the mark; TRANSFER to a 50 mL plastic beaker. Under magnetic stirring, measure the potential value. From the standard curve, the corresponding fluorine content is obtained, to calculate the soluble fluorine content.

5.8.6 Determination of free fluorine

Accurately pipette 2.0 mL of the supernatant prepared in 5.8.3 into a 50 mL plastic volumetric flask. ADD 5 mL of citrate buffer; USE deionized water to dilute to the mark; TRANSFER to a 50 mL plastic beaker. Under magnetic stirring, measure the potential value. From the standard curve, the corresponding fluorine content is obtained, to calculate the free fluorine content. If the free fluorine content in the sample is too high, according to the actual situation, the sampling quantity may be appropriately diluted or reduced.

5.8.7 Calculation formula

According to formula (1), calculate the soluble fluorine or free fluorine content (X) in the sample:

$$X = \text{antilog}c \times (50/2.0) \times (100/m) \qquad \cdots\cdots(1)$$

Where:

X - Soluble fluorine or free fluorine content, in milligrams per kilogram (mg/kg);

c - Fluorine content in the test solution, in milligrams per kilogram (mg/kg);

m - Sample mass, in grams (g).

Finally, the above calculation result unit (mg/kg) is converted into a percentage concentration and is accurate to two digits after the decimal point.

5.8.8 Allowable difference

The allowable difference of the results of the two parallel determinations is ±5%.

5.9.4.1 Temperature programming: Initial temperature is 60 °C. Initial time is 1.8 min. Heating rate is 40 °C/min. Final temperature is 160 °C, hold for 3 min.

5.9.4.2 Inlet temperature: 200 °C.

5.9.4.3 Detector (FID) temperature: 300 °C.

5.9.4.4 Carrier gas: Nitrogen, 42.3 kPa.

5.9.4.5 Split ratio: 1+13.

5.9.4.6 Septum purge: 2 mL/min.

5.9.4.7 Make-up gas: Nitrogen, 25 mL/min.

5.9.5 Preparation of fluoride ion standard solution and internal standard solution

5.9.5.1 Preparation of fluoride ion standard solution

Prepare according to 5.8.2.4.

5.9.5.2 Preparation of internal standard solution

WEIGH about 0.5 g of n-pentane in a 100 mL glass volumetric flask; USE toluene to dilute to the mark and shake well. This internal standard stock solution is stored in the refrigerator and is valid for 1 month.

PIPETTE 5.0 mL of the internal standard stock solution into a 250 mL glass volumetric flask; USE toluene to dilute to the mark and shake well. This internal standard solution must be re-prepared each time.

During the same analysis, all standard solutions and sample solutions must be prepared with the same internal standard solution.

5.9.6 Preparation of standard solution and sample solution

5.9.6.1 Preparation of standard solution

PIPETTE 1.0 mL and 5.0 mL of fluoride ion standard solution into 2 40 mL plastic bottles with caps; ADD 10% sodium chloride solution to make the total volume 10 mL; obtain standard solution A and solution B. The fluoride ion masses are 0.1 mg and 0.5 mg, respectively.

5.9.6.2 Preparation of sample solution

WEIGH approximately 0.2 g of sample (accurate to 0.0001 g) into a 40 mL plastic bottle with cap. PUT 3 glass beads in; ADD 10.0 mL of 10% sodium

According to formula (3), calculate the total fluorine content (Z) in the sample:

$$Z = \frac{A_3}{m \times K_M \times A_4} \times 100\% \quad \cdots\cdots (3)$$

Where:

Z - The total fluorine content in the sample. The result is accurate to two digits after the decimal point, %;

A_3 - The peak area of the fluoride in the sample solution;

A_4 - The peak area of n-pentane in the sample solution;

m - Sample mass, in milligrams (mg).

The detection limit of this method is 0.2 mg/kg. The lower limit of quantitation is 1 mg/kg.

5.10 Determination of net quantity

It shall be carried out according to JJF 1070.

6 Inspection rules

6.1 Inspection classification

6.1.1 Type inspection

Toothpaste type inspection includes all items in the standard. During normal production, it shall not be less than one time per quarter. In one of the following cases, type inspection shall also be carried out:

a) When there is a significant change in one of raw materials, processes, and formulations and the product performance may be affected;

b) When the production resumes after the product is discontinued for a long time;

c) When the exit-factory inspection result of the product is significantly different from the previous type inspection;

d) When the national quality supervision agency proposes a requirement for type inspection.

6.1.2 Exit-factory inspection

7 Marking

7.1 The sales packaging and maximum surface area calculation method of the product shall be carried out in accordance with the provisions of GB 29337.

7.2 Large packaging shall have the following marks:

a) Product name;

b) Producer's name and plant site: The producer's name and address shall be the name and address of the producer who is legally registered and can bear the responsibility for product quality;

c) Packing quantity, gross weight;

d) Production date or restricted use date and production lot number;

e) Packaging case volume [length (mm)×width (mm)×height (mm)];

f) Pictorial marking for handling of package: The enterprise shall, based on the actual requirements of the product, according to the provisions of GB/T 191, indicate pictorial marking for handling of large package.

8 Transportation and storage

8.1 Transportation

It must be lightly loaded and unloaded and stacked according to box arrows. It shall avoid severe shake, impact, and sun and rain.

8.2 Storage

It shall be stored in a ventilated and dry warehouse. It shall not be close to the water source and heating. When stacking, it shall be more than 10 cm away from the ground and be more than 50 cm away from the wall, leaving the passage. It shall be stacked according to the box arrows and shall not be placed upside down.

magnetic stirrer (about 20 min), until it becomes a homogeneous solution.

A.3 Sample determination

Under the condition that the sample solution treated by A.2 is stirred on a magnetic stirrer, use a microinjector to pipette 10 μL (do not pipette bubbles); immediately inject into a graphite tube; START the graphite furnace switch to measure; RECORD the absorption value. Also make a blank test.

A.4 Making of standard curve

According to the absorption value of the sample, prepare 3~5 lead standard solutions with the corresponding concentrations; measure the absorption values. The blank absorption value is subtracted, to prepare a concentration and absorption curve.

A.5 Calculation

The blank absorbance value is subtracted from the sample absorbance value. According to the standard curve, obtain the corresponding concentration, which is multiplied by the dilution factor, to obtain the lead content in the sample toothpaste.

According to formula (A.1), calculate the lead content (w) of the sample:

$$w = k \times c \qquad \cdots\cdots (A.1)$$

Where:

w - The lead content of sample, in milligrams per kilogram (mg/kg);

k - The dilution factor of sample;

c - The corresponding concentration obtained from the standard curve, in milligrams per kilogram (mg/kg).

A.6 Allowable difference

The allowable difference of the results of the two parallel determinations is ±5%.

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