GB

National Standard of the People's Republic of China

GB8372 – 200x
Replacing GB8372-2001

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Toothpaste

(Draft for Approval)

Issue date: 200×-××-××   Implementation date: 200×-××-××

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Forward

The indicators of odour, paste squeeze pressure and foam volume set out in Table 4.2, packaging in Subsection 7.2, and transportation in Subsection 7.3 are recommendatory clauses. All other clauses are mandatory.

This Standard refers to the international Standard ISO11609: Dentistry — Toothpastes — Requirements, test methods and marking 1995 (E).

This Standard replaces GB8372-2001, "Toothpaste".

Compared with GB8372-2001, the main changes to this Standard are as follows:

— the scope of application of the Standard has been verified;

— the terms and definitions have been increased;

— it is specified that the raw materials used in the manufacture of toothpaste should meet "Requirements for toothpaste- use raw materials";

—the requirements of the total number indices of mould and yeast have been increased;

— it is specified that the micro-organism index test should conform to the requirements of "Hygienic Standard for Cosmetics";

— requirements regarding foam volume index of children's toothpaste have increased;

— the pH index has been revised;

— requirements regarding total fluorine volume index have been revised;

— requirements regarding soluble fluorine or free fluorine volume index have been revised;

— requirements regarding the total fluorine volume index of children's fluoride toothpaste have been revised;

— requirements regarding soluble fluorine or free fluorine volume index of children's fluoride toothpaste have been revised;

— it is specified that net contents should be finished according to the Order No 75 [2005] of the General Administration of Quality Supervision, Inspection and Quarantine, the People's Republic of China, "Measurement, supervision and management methods of quantitative packed products";

— part of the marking, packaging and storage requirements has been revised.

Appendix A to this Standard is a standardised appendix.

This Standard was proposed by the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China.

The organisation in charge of this Standard is the National Toothpaste and Wax Products Standardisation Centre.

The main drafting unit for this Standard is the General Administration of Quality Supervision, Inspection and Testing of Toothpaste and Wax Product Light Industry.

The main drafters of this Standard are: Sun Dongfang, Ma Xuan, Li Xianbo, Shen Erui, and Meng Yu.

Previous versions of this Standard to be replaced by this Standard are:

— QB592-81, GB8372-87, GB8372-1995, and GB8372-2001.

As from the date of implementation, this Standard replaces the original national Standard GB8372-2001 "Toothpaste", implemented by the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China.

Toothpaste

1   Scope

This Standard sets the terms and definition, requirements, test methods, inspection rules, marking, packaging, transportation and storage of toothpaste.

This Standard applies to different kinds of toothpaste for cleaning and caring for the mouth.

2   Normative references

The provisions of the following documents become provisions of this Standard after being referenced. For dated reference documents, all later amendments (excluding corrigenda) and versions do not apply to this Standard; however, the parties to the agreement are encouraged to study whether the latest versions of these documents are applicable. For undated reference documents, the latest version apply to this Standard.

GB 7917.2   Standard methods of hygienic test for cosmetics Arsenic.

GB 191   Packaging — Pictorial marking for handling of goods (EQV ISO 780: 1997).

GB/T 601   Chemical reagent — Preparations of standard volumetric solutions.

GB/T 602   Chemical reagent — Preparations of stock standard solutions (NEQ ISO 6353-1: 1982).

GB/T 603   Chemical reagent — Preparations of reagent solutions for use in test methods (NEQ ISO 6353-1: 1982).

GB/T 6682   Water for analytical laboratory use — specification and test methods (NEQ ISO 3696: 1987).

GB/Z ××××-200× Requirements for toothpaste-use raw materials.

No 172 [1997] issued by the National Quality and Technical Supervision Bureau, "Provisions on indicating product identification."

Order No 75 [2005] of the General Administration of Quality Supervision, Inspection and Quarantine, the People's Republic of China, "Measurement, supervision and management methods of quantitative packed products."

JJF1070-2005   Measurement and inspection rules for net contents of quantitative packaged products.

"Hygienic standard for cosmetics," 2007 Version issued by the Ministry of Health of the People's Republic of China.

3   Terms and definitions

This Standard adopts the following terms and definitions.

3.1

Toothpaste

A substance in paste form mixed with and composed of friction agent, moisturiser, thickening agent, foaming agent, aromatics, water and other additives (including ingredients for improving the health conditions of the mouth).

4   Requirements

The raw materials used for producing toothpaste should meet the "Requirements of toothpaste-use raw materials."

4.1   Hygiene indices

Hygiene indices should meet the requirements set out in Table 1.

4.2   Sensory, physical and chemical indices

Sensory, physical and chemical indices should meet the requirements set out in Table 2.

4.3   Net contents

The net contents of toothpaste should conform to Order No 75 [2005] of the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China, "Measurement, supervision and management methods for quantitative packaged products."

Table 1

Item			Requirement
Micro-organism Index	**Total number of bacterial colony (CFU/g)**	•	500
	Total number of mould and yeast (CFU/g)	•	100
	Faecal coliform group / g		Nil
	Pseudomonas aeruginosa / g		Nil
	Staphylococcus aureus / g		Nil
Toxic substance limit	**Heavy metal (in Pb) content / (mg/kg)**	•	15
	Arsenic (As) content / (mg/kg)	•	5
Remarks: The words set in bold characters are mandatory indices.			

Table 2

Item		Requirement
Sensory Index	**Paste**	**Clean, even, tight, normal colour**
	Odour	Meet the specified odour type

Physical and Chemical Indices	Thickness / mm		9 ~ 33
	Paste squeeze pressure / kPa	•	40
	Foam volume / mm	•	60
		•	120 (applicable to children's toothpaste)
	pH value		5.5 ~ 10.0
	Stability		The paste does not leak out of the tube, no liquid is separated from the paste, odour and colour are normal.
	Excessively hard grain		No scratches are found on the glass.
	Total fluorine vol./%		0.05~0.15 (applicable to fluoride toothpaste) 0.05~0.11 (applicable to children's fluoride toothpaste)
	Soluble fluorine or free fluorine vol./%		0.05~0.15 (applicable to fluoride toothpaste) 0.05~0.11 (applicable to children's fluoride toothpaste)
Remarks: The words set in bold characters are mandatory indices.			

4.4 Requirements of packaging appearance

4.4.1 **Pliable tube or other packaging**

4.4.1.1 The tube body is clean-cut, neat, firmly welded, and undamaged. Images and words are printed clearly and completely. The colour process is accurate, without any obvious printing defects or ink loss;

4.4.1.2 The cap is clean, neat, with even colour, and without fading. The cap and the thread at the tube mouth are tightly matched, but not loosened or stripped. The part of the easy-to-open cap that is lifted is firmly connected to the body of the cap, and tightly sealed up, without any disclosure of the paste;

4.4.1.3 The tube end is firmly sealed without leakage;

4.4.1.4 If there is a sealing film at the mouth, it is clean, complete, and can be torn off easily.

4.4.2 **Box (applicable to toothpaste packaged in small boxes)**

4.4.2.1 The surface of the box is clean-cut, neat, with patterns printed clearly and with accurate colour process.

4.4.2.2 The body of the box should be flat, has no cracks and no delaminating.

5 Test methods

Unless otherwise specified, both the reagents and water used in this Standard are analytical reagents and water which meet the requirements set out in GB/T6682.

Unless otherwise specified, the standard solution for use in the analysis, the

solution for impurity determination, the making agent and products for test methods should be prepared according to the requirements set out in GB/T601, GB/T602 and GB/T603.

Regarding the calculation of the index of the final test result according to the method of weighing, 20 mm of the paste should be firstly squeezed out and discarded. After that, squeeze out the weighted volume as the sample.

5.1 **Micro-organism index**

Inspection should be carried out according to the methods set out in "Hygienic Standard for Cosmetics."

5.2 **Heavy metal (counted in Pb) content**

This method belongs to arbitration inspection method. The non-arbitration inspection can be based on the method specified in the normative Annex A.

5.2.1 **Toothpaste containing calcium carbonate and calcium phosphate as basic substances**

5.2.1.1 Reagents

a) Nitric acid;

b) Nitric acid solution: 5 mol / L;

c) Nitric acid solution: 0.2 mol / L;

d) Nitric acid solution: 0.01 mol / L;

e) Hydrogen peroxide solution content: 30%;

f) Ammonia water: ammonia content 25%~28%;

g) 1% ammonium hydroxide solution: add water to 4ml of ammonia water to a dilution of 100 ml;

h) 10% ammonium sulphamate solution: weigh and take 10 g of ammonium sulphamate. Add water to dilution to 100 ml;

i) 2% APDC solution: APDC (ammonium pgrroline dithiocarbamate) is the atomic absorption analytical reagent. Weigh and take 500 mg of APDC. Add 25 ml of water and dissolve. The solution must be poured into a brown bottle, which is then stored in the refrigerator. One week later, a re-blend shall be made;

j) Standard lead reserve liquid: contain 1 mg/ml of lead;

k) Standard lead solution: absorb 10.0 ml of lead reserve liquid into a 100 ml volumetric flask. Dilute it by 0.01 mol/l of nitric acid solution until reaching the graduation (contain 100• g/ml of lead). Separate the dilution using 0.01 mol/l of nitric acid solution, making lead become the standard solutions at 1• g/ml, 3• g/ml and 5• g/mL;

l) Chloroform;

m) Hg^{2+} solution: weigh and place 0.537g of mercuric oxide into a small beaker, and then add 1ml of nitric acid to dissolve it. Add around 50ml of water, and filter it.

Use some water to clean the beaker and funnel. Add around 480ml of water. Use 5mol/l and 0.2mol/l of nitric acid solution to adjust the pH to 1.6. Add water to 500ml. The solution contains 1,000• g/ml of Hg^{2+}.

5.2.1.2 **Instruments**

a) pH meter: accuracy at 0.02pH. Use buffer solution pH=4.00 for calibration;

b) Atomic absorption spectrophotometer: at a wavelength of 283.3nm.

5.2.1.3 **Preparation and determination of sample**

Take a tube of toothpaste at random to use as the sample. Weigh 2g of toothpaste, and accurately place 0.01g into a 150ml Erlenmeyer flask. Add 5ml of water and 5ml of nitric acid. Heat the flask under a low flame and shake until the paste inside is dissolved. After it is gradually cooled down, add in 1.5ml of 30% hydrogen peroxide solution, and shake the flask. Heat under a low flame until the hydrogen peroxide is completely broken down. If any nitrogen dioxide smoke appears in a brick red colour, immediately add 2ml of 10% ammonium sulphamate solution and heat until the solution is slightly boiling. Rapidly add water to make up to 50ml so that it cools down quickly. Add 3ml of ammonium water. After this has cooled down to room temperature, pour the solution into a 100ml beaker. Use 10ml of water to clean the Erlenmeyer flask twice. Use ammonium water and 1% ammonium hydroxide solution to adjust the pH to 1.1 ~ 1.2. During this time, the solution will appear to be slightly turbid. Filter this though a 125ml separator funnel. Use 5ml of water to clean the beaker and funnel. 1ml of 2% APDC solution is added to the solution. Shake the flask evenly. Allow it to stay in quiescence for 3 minutes. Add 10ml of chloroform, and shake the flask for 2 minutes. After layering, the chloroform is transferred to another separator funnel. Again, use 10ml of chloroform to repeat the extraction. Combine the extracted solutions. Add 10.0ml of Hg^{2+} solution, and shake the flask for 2 minutes. After layering, the upper-layer water phase is supplied to conduct the determination of flame atomic absorption. Meanwhile, take 0.01 mol/l of nitric acid as being blank. Determine the absorption of the standard lead series: 1• g/ml, 3• g/ml and 5• g/ml. Using lead concentration as the horizontal coordinate and lead absorption as the vertical coordinate, draw a standard curve.

5.2.2 **Toothpaste with aluminium hydroxide and silica as the basic substances**

5.2.2.1 **Reagents**

Sulphuric acid.

Other reagents are the same as Subsection 5.2.1.1.

5.2.2.2 **Instruments**

The same as Subsection 5.2.1.2.

5.2.2.3 **Preparation and determination of samples**

a) Aluminum hydroxide toothpaste: take a tube of toothpaste at random as the

sample. Weigh 2g of toothpaste, and accurately place 0.01g into a 250ml Erlenmeyer flask. Add 15ml of nitric acid and 1ml of sulphuric acid and heat until nitrogen dioxide gas is produced in a brick red colour. Take down the flask and allow it to cool down for a while. Add 2ml of hydrogen peroxide solution, shake the flask and allow it to cool down to room temperature. Add 10~15ml of water and 1ml of hydrogen peroxide solution and boil for 5~6 min. While boiling, the flask should be shaken continuously to remove the hydrogen peroxide. Add in 2ml of 10% ammonium sulphamate solution. After it cools down slightly, take it down. Rapidly add water to 60ml, and allow it to cool down to room temperature. After the pH value of the solution is adjusted to 1.0, move it to a 125ml separator funnel.   Add in 1mL of 2% APDC solution and 10ml of chloroform, and shake the flask for 2 min. After layering, chloroform is transferred to another separator funnel. Again, use 10ml of chloroform to repeat the extraction. Combine the extracted solutions. Add 10.0ml of Hg^{2+} solution, and shake the flask for 2 min. After layering, the upper-layer water phase is supplied to conduct the determination of flame atomic absorption. Meanwhile, take 0.01 mol/l of nitric acid as being blank. Determine the absorption of the standard lead series: 1•g/ml, 3•g/ml and 5•g/ml. Use lead concentration as the horizontal coordinate and lead absorption as the vertical coordinate to draw a standard curve.

b) Silica toothpaste: take a tube of toothpaste at random as the sample. Weigh 2g of toothpaste, and accurately place 0.01g into a 250mL Erlenmeyer flask. Add 5ml of water and 5ml of nitric acid and heat under a low flame until the paste is dissolved. After it has gradually cooled down, add 1.5ml of hydrogen peroxide solution, and shake the flask. Heat under a low flame until hydrogen peroxide gas in a red brick colour is created. Add 2ml of 10% hydrogen peroxide solution. After it has been slightly heated, take it down. Add 20ml water to cool it down to room temperature. Use two layers of filter paper to conduct filtering. Use 15ml of water and clean the Erlenmeyer flask several times, the inner wall of the Buchner funnel, and clear the sediments. The filtered solution is poured into the 100ml beaker. Use 10ml of water to clean the filtering flask twice. Adjust the pH value to 1.2. Regarding the following operation, with the exception of the addition of 5.0ml of Hg^{2+} solution for back extraction, the rest of the complexing, extraction and determination are carried out by aluminium hydroxide toothpaste.

5.2.3 **Admissible error**

The admissible error between the results acquired from two times of parallel determination is ± 5%.

5.3 **Silicon content**

According to the silicon speck inspection method of GB7919.2.

5.4 Paste

Take two tubes of toothpaste at random as the samples. Dissect the tube, and carry out a visual test according to the index requirements.

5.5 Odour

Identify the odour by smelling.   Identify the flavour by using tongue-tasting method.

5.6 Thickness

5.6.1 **Instruments**

5.6.1.1 Thermostat: Accuracy at $\pm$ 2°C;

5.6.1.2 Stop watch;

5.6.1.3 Standard cap: there is a small hole at a diameter of 3mm at the centre. The inner diameter and thread of cap should be consistent with the corresponding tube type of toothpaste;

5.6.1.4 Thickness measurement shelf: the rectangular metal shelf is installed with 13 stainless steel wires at a diameter of 1.5mm. The first steel wire is installed at the end of the metal shelf. The central distance between the second and first wires is 3mm, and the distance between the third and second wires is 6mm. After that, for each increase of a stainless steel wire, the distance is increased by 3mm, until the total length of 36mm. The distance figure of each stainless steel wire is just the reading of the thickness. The first wire is 0, the second wire is 3, the third wire is 6, the fourth wire is 9, and so on and so forth.

5.6.2 **Determination procedures**

5.6.2.1 Place 3 samples of toothpaste tubes in a thermostat at 45°C $\pm$ 2°C.   Then, place another 3 samples of toothpaste tubes at room temperature. After 24 hours, these tubes of toothpaste are to be determined.

5.6.2.2 The samples to be determined are sealed by standard caps. Discard the first 20mm of toothpaste squeezed out. Allow the paste to cross over the steel wires in fine threads on the thickness shelf from the first one to the rest of them. When the paste is squeezed out, the paste in fine threads should hang vertically on the steel wires, and the squeezing speed should be even and consistent. From each tube of toothpaste, three threads of paste are squeezed out continuously. After squeezing, allow the paste to remain hanging for 1 min., and observe the falling situation of the paste.

5.6.2.3 **Indication of results**

a) Among the three paste dangles, the greatest distance in mm between 2 unbroken paste dangles both horizontally crossing over the steel wires is considered the determination result of a single tube of toothpaste. If the thickness of the three hanging paste threads is different, the middle value is taken as the determination result of the single tube of toothpaste.

b) After three tubes of toothpaste have been determined, two of them having the

same thickness are taken as the final results. If the determination results of three tubes of toothpaste are different, the middle value is taken as the final result. If the determination result is smaller than 9mm or greater than 33mm, the toothpaste is judged as failed; and if it is at the range of 9~33mm, the toothpaste is judged as passed.

5.7 **Paste squeeze pressure**

5.7.1 **Instruments**

5.7.1.1 Refrigerator: accuracy at $\pm 1^{o}C$;

5.7.1.2 Determined paste squeeze pressure;

5.7.1.3 Condensation pump;

5.7.1.4 Pressure gauge: accuracy at 1 Pa, measurement range at 0~40kPa;

5.7.1.5 Standard cap: the same as Subsection 5.6.1.3.

5.7.2 **Determination procedures**

Take two tubes of toothpaste at random as the samples. Place them in a refrigerator at $-8^{o}C \pm 1^{o}C$ for 8 hours, and then remove them. Use hand to squeeze out around 20mm of toothpaste and discard it. The mouth of the toothpaste tube is screwed into the standard cap of the paste squeeze pressure determiner. Then, tightly screw the standard cap together with the toothpaste into the air cylinder of the paste squeeze pressure determiner, and no air should leak out. Slowly press air into the air cylinder through the condensation pump. When 1mm~2mm of the paste is squeezed out, stop the air supply. Open the exhaust piston of the air cylinder, and let the pressure gauge resume to zero. Use a small knife to scrape the mouth of the pliable tube to strike off the paste squeezed out, and close the exhaust piston. Press in air again. When 1mm~2mm of paste is squeezed out, record the pressure reading on the pressure gauge immediately. The absolute difference of the parallel determination values should not be greater than one. Take the greatest determination value as the determination result.

5.8 **Foam volume**

5.8.1 **Instruments**

5.8.1.1 Super thermostatic water bath: Accuracy at $\pm 1^{o}C$;

5.8.1.2 Ross-Miles foam determiner.

5.8.2 **Preparation of test fluid**

Take a tube of toothpaste at random as the sample. Weigh and take 10g of toothpaste from it, and accurately place 0.01g into a 100ml beaker. Meanwhile, add 100ml of distilled water for heating to $40^{o}C \pm 1^{o}C$. First of all, use a small amount of distilled water to blend the sample to be in thick liquid state. Pour the sample in thick liquid state into a 1,000ml porcelain cup, and clean the beaker several times. Finally, pour the remaining distilled water into the 1,000ml porcelain cup, and stir it

slightly and evenly. Pour into a $40^{o}C \pm 1^{o}C$ thermostatic water bath to keep it warm, and then wait for the results.

5.8.3 Determination procedures

5.8.3.1 Preparation work

Turn on the super thermostatic water bath circulation device that connects with the graduated tube jacket of Ross-Miles foam determiner, and keep the foam determiner at $40^{o}C \pm 1^{o}C$. First of all, use a clean and well-prepared drip tube to hold 200ml of distilled water at $40^{o}C \pm 1^{o}C$ to clean the inner wall of the graduated tube. Then, use the test solution to wash along the wall. The washing should be complete. Pour the test solution being preheated to $40^{o}C \pm 1^{o}C$ to the lower end of the graduated tube, and adjust the surface of test solution to the graduation of 50ml.

5.8.3.2 First test

Use the drip tube to absorb accurately 200ml of test solution. Next, place it on the graduated tube carrier and allow it to be vertical to the cross-section of the graduated tube, making the test solution reach the central position of solution surface inside the graduated tube when being poured into the tube. Open the piston of the drip tube, and let the test solution drip down once. Upon the dripping of the test solution, immediately record the high and low peak values of foam. Calculate the average mathematical value of two peak values, and take it as the test result of this time.

5.8.3.3 Second test

Repeat the procedures of Subsections 5.8.3.1 and 5.8.3.2, and carry out the second test.

5.8.3.4 Calculation results

The absolute difference between the test results of the first and second tests should be smaller than 5mm. Take the average mathematical value of the two test results as the determination result.

5.9 pH value

5.9.1 Instruments

5.9.1.1 pH meter: Accuracy at 0.02pH unit;

5.9.1.2 Thermometer: Accuracy at $0.2^{o}C$;

5.9.1.3 Balance: Accuracy at 0.01g.

5.9.2 Determination procedures

Take a tube of toothpaste at random as the sample. Weigh and take 5g of toothpaste from the tube, and accurately place 0.01g into a 50ml beaker. Add 20ml of the pre-boiled and cooled distilled water for stirring thoroughly and evenly, and determine it by the pH meter under $20^{o}C$.

5.9.3 Indication of results

The absolute difference between the parallel determination values should not be

greater than 0.2pH unit. Take the average mathematical value as the determination result.

5.10 **Stability**

5.10.1 **Instruments**

5.10.1.1 Refrigerator: accuracy at ± 1°C;

5.10.1.2 Electro-heating standing-temperature cultivator: accuracy at ± 1°C.

5.10.2 **Reagent**

Filter paper: 102 at medium speed.

5.10.3 **Determination procedures**

Place a toothpaste sample in a refrigerator at -8°C ± 1°C for 8 hours. Then, remove it and place it in a standing-temperature cultivator at 45°C ± 1°C. After 8 hours, remove it and allow it to reach room temperature again. Open the cap, and the paste should not leak out of the tube mouth. Squeeze out all the paste on a white filter paper. No liquid should be separated from the paste, and the odour and colour should be normal.

5.11 **Excessively hard grain**

5.11.1 **Instruments**

5.11.1.1 A determiner of excessively hard grain;

5.11.1.2 Microslide: 75mm× 25mm.

5.11.2 **Determination procedures**

Take a tube of toothpaste at random as the sample. Weigh and take 5g of toothpaste on a microslide without any scratch mark.　Place the microslide into the fixed tank of the determiner.　Press on the friction copper billet, and turn on the switch. After the copper billet has undergone forward and backward motion friction 100 times, remove the friction and take out the microslide. Use water or 1+1 hot nitric acid to clean the microslide, and then observe whether any scratches appear on it.

5.12 **Instruments**

5.12.1.1 Ion meter:　Attached with fluoride ion-selective electrode and reference electrode. The division value of potential measurement should not be greater than 0.2mV;

5.12.1.2 pH meter:　Accuracy at 0.02 pH;

5.12.1.3 Ion precipitator;

5.12.1.4 Diffusion chamber (see Figure 1).

5.12.2 **Reagents**

5.12.2.1 Hydrochloric acid solution: 4 mol/l;

5.12.2.2 Sodium hydroxide solution: 4 mol/l;

5.12.2.3 Potassium hydroxide solution: 2 mol/l;

5.12.2.4 Perchloric acid solution: 0.4 mol/l;

5.12.2.5 Hexamethyl disiloxane saturated perchloric acid solution: Pour 58% perchloric acid solution into a separator funnel, and add in hexamethyl disiloxane (HMDS). After drastic shaking of the funnel, let it stay in quiescence for layering. The lower layer is just the hexamethyl disiloxane saturated perchloric acid solution;

5.12.2.6 Citrate buffer solution: 100g of trisodium citrate, 60mL of glacial acetic acid, 60g of sodium chloride, and 30g of sodium hydroxide are dissolved by water. Adjust the pH value to be 5.0~5.5, and use water to dilute it to be 100mL;

5.12.2.7 Fluoride ion standard solution: Accurately weigh and take 0.1105g of standard sodium fluoride (drying at $105^{o}C \pm 2^{o}C$ for 2 hours). Use deionised water to dissolve it, and obtain a predetermined volume to 500ml. Shake it and store in a polyethylene plastic flask for later use The concentration of the solution is 100mg/kg.

5.12.3 **Preparation of sample**

Take a tube of toothpaste at random as the sample. Weigh and take 20g of toothpaste, and accurately place 0.01g into a 50ml plastic beaker. Gradually add in deionised water to stir it until it is dissolved. Transfer it to a 100ml plastic volumetric flask. Dilute it until reaching the graduation, and shake it. Pour it into two separate graduated 10ml centrifuge tubes, at equal weight. After being centrifuged in the centrifuge machine (2000r/min.) for 30 min., it is cooled down to room temperature. Its supernatant liquid is used for the analysis of free fluorine and the concentration of soluble fluorine, whereas its suspension is for the analysis of the concentration of total fluorine.

5.12.4 **Drawing of standard curve**

Accurately absorb 0.5, 1.0, 1.5, 2.0 and 2.5mL of fluoride ion standard solution, and move them into five 50ml plastic volumetric flasks. 5ml of citric buffer solution is added to each of them. Use deionised water to dilute it until reaching the graduation. After this, each solution is poured in a 50ml plastic beaker. When the solutions are being stirred by magnetic force, measure the potential values E, record them, and draw an E-logC (C is the concentration) standard curve.
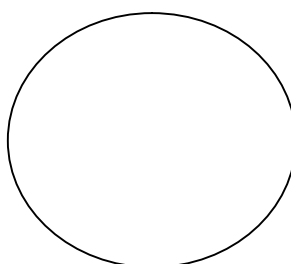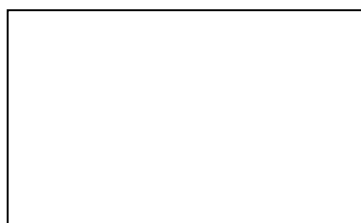
1 – diffusion chamber cover   2 – diffusion chamber   3 – absorption pool

Diffusion chamber cover: $110mm \times 20mm$

Diffusion chamber: $d110$ mm $\times 60mm$

Absorption pool: $L \times S \times H = 75mm \times 50mm \times 35mm$

Figure 1   Diffusion chamber

5.12.5 Determination of free fluorine

Absorb 10ml of supernatant liquid, and place it in a 50ml plastic volumetric flask.   Add in 5ml of citric buffer solution. Use deionised water to dilute it until reaching the graduation. Transfer it to a 50ml plastic beaker. When it is being stirred by a magnetic force, measure its potential value. On the standard curve, check its corresponding fluorine content, and then calculate the concentration of free fluorine.

5.12.6 Determination of soluble fluorine

Absorb 0.5ml of supernatant liquid, and transfer it into a 2mL of micro centrifuge tube. Add 0.7ml of 4 mol/l hydrochloric acid. Cover the centrifuge tube. Put it in water bath at $50^{o}C$ for 10 min. Transfer it to a 50ml volumetric flask. Add 0.7ml of 4mol/l sodium hydroxide for neutralising.   Then, add in 5ml of citrate buffer solution. Use deionised water to dilute it until reaching the graduation. Transfer it to a 50ml plastic beaker. When it is being stirred by magnetic force, measure its potential value. On the standard curve, check its corresponding fluorine content, and then calculate the concentration of soluble fluorine.

5.12.7 Determination of total fluorine content

5.12.7.1 The connecting place between the diffusion chamber and the chamber cover is applied with Vaseline. The chamber is placed at an inclined position (see Figure 2), forming three small chambers separating from each other, as shown in Figure 2. Absorb 0.5ml of suspension and transfer it to the left chamber of the diffusion chamber.   Add in 5ml of 0.4 mol/l of perchloric acid solution, and stir it for a while. Add 0.5ml of 2mol/l potassium hydroxide.   Transfer 5ml of hexamethyl disiloxane saturated perchloric acid solution to the right chamber.   Seal the diffusion chamber tightly by the cover.   Thoroughly blend the solutions of the left and right chambers, and let it stay in quiescence for above 7 hours.

5.12.7.2 Open the diffusion chamber. Remove the solution of the central chamber. Transfer the solution to a 50ml volumetric flask. Add 0.25ml of 4 mol/l hydrochloric acid for neutralizing. Add in 5ml of citrate buffer solution. Use deionised water to dilute it until reaching the graduation. Transfer it to a 50ml plastic beaker. When it is being stirred by magnetic force, measure its potential value. On the standard curve,

check its corresponding fluorine content, and then calculate the concentration of total fluorine.

5.12.8 Calculation formulas

Free fluorine = antilog $c \times (50 / 10) \times (100 / m)$ …………………. (1)

Soluble fluorine = antilog $c \times (50 / 0.5) \times (100 / m)$ ………..…….. (2)

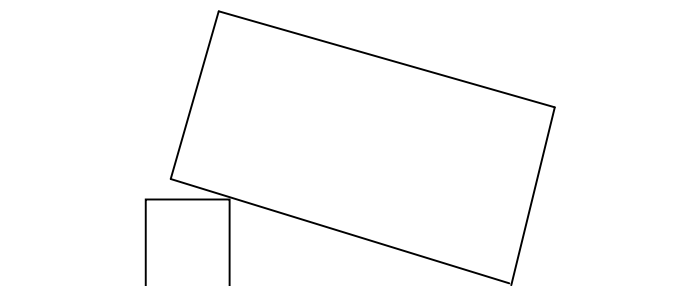Total fluorine = antilog $c \times (50 / 0.5) \times (100 / m)$ …………..…….. (3)

Figure 2   Placement of diffusion chamber

Remarks: Material of diffusion chamber: polystyrene

In the formulas: antilog c — anti-logarithm value, which is acquired from the logarithm value of fluorine content found on the standard curve

m — mass of sample, in grams.

Finally, the above calculation results in mg/kg are converted to percentage concentration.

5.12.9 For the statement, "Absorb 0.5ml of suspension" in Subsection 5.12.7.1, the suspension can also be replaced by 0.2g~0.3g of paste being added with small volume of deionised water. Its calculation equation is:

Total fluorine = antilog $c \times (50 / m)$ …………..…………………….. (4)

5.12.10 Admissible error

The admissible error between the parallel determination results of the two is $\pm$ 5%．Their average arithmetic value is taken as the determination result.

5.13 Determination of net contents

Carry out the inspection according to methods set out in JJF1070.

6 Inspection rules

6.1 Inspection categorisation

6.1.1 Type inspection

The inspection of toothpaste type includes all the items of this Standard. Type inspection should be carried out at least once every season of normal production. Under one of the following circumstances, type inspection should also be carried out:

6.1.1.1 When the technical conditions or prescription of raw material has any

tremendous change that may affect the performance of product;

6.1.1.2 When a product resumes its production after suspended production for a long time;

6.1.1.3 When there is a greater difference between the ex-factory inspection result and the type inspection of the last time;

6.1.1.4 When the national quality supervision institution has the request of type inspection.

6.1.2 **Ex-factory inspection**

Before a product is delivered from the factory, it must pass the inspection by the production factory. Ex-factory inspection items are physical and chemical indices, sensory index, as well as the requirements of net contents and package appearance specified in Section 4.

6.2 **Batch and sampling, judgment rules**

6.2.1 Toothpaste should be delivered batch by batch. Products of the same technical conditions, variety, specifications and production date are considered to be a batch. The consignee can also take the delivered products for once as a batch.

6.2.2 **Sampling**

Take carton as the unit. Confirm the samples according to the size of batch and based on Table 3.

Table 3

Batch (No. of Cartons)	Sampling (No. of Cartons)
• 500	5
501 ~ 1,000	8
> 1,000	13

Randomly take cartons of samples at the delivery place or the warehouse of the finished products of toothpaste. When inspecting the packaging quality, inspect all the small packages inside the carton, and carry out the inspection according to Subsection 4.4.2. The total judgment rate of the passed products is 10%. After this, take 2~4 medium boxes at random from each carton, and then take the same number of tubes from each medium box, making the total number of samples to be no less than 40 tubes.

6.2.1.1 If the inspection is not passed, samples can be taken out from 2 times of the carton samples to carry out re-inspection of the failed items. If the re-inspection results are still failed, this batch of products is regarded as being failed.

6.2.1.2 When the consignor and consignee have argument over the inspection result, arbitration inspection can be carried out, and the arbitration result is the final reference.

7   **Marking, packaging, transportation, storage**

7.1 Marking

7.1.1 The package for sale should contain the following marking:

7.1.1.1 Name and brand of product;

7.1.1.2 Net contents;

7.1.1.3 Name and address of production factory;

7.1.1.4 Execution standard number;

7.1.1.5 Guarantee period and production date or expiry date and production batch number;

7.1.1.6 Odour type;

7.1.1.7 The ingredients of the toothpaste should be indicated with the standard names of the raw materials. Fluoride toothpaste should be indicated with the amount of added fluorine. Children's fluoride toothpaste should be indicated with warning words.

7.1.1.8 The marking of travellers' toothpaste should be according to No. 172 [1997] issued by National Quality and Technical Supervision Bureau, "Provisions on indicating product identification." Regarding the travellers' toothpaste with a marketed package at a net weight of less than or equal to 10g, the package can be indicated with only the name, brand, net contents, name of production factory, guarantee period and production date or expiry date and production batch number, with no need to indicate the odour type, adopted standard number, and so on.

7.1.2 **Large package should include the following marking:**

7.1.2.1 Name of product;

7.1.2.2 Name and address of production factory;

7.1.2.3 Number of cartons, gross weight;

7.1.2.4 Production date or expiry date and production batch number;

7.1.2.5 The specifications of packaging carton should meet the requirements of GB/T191;

7.1.2.6 Identifications such as "Upward," "no rainfall," "no sunshine," and so on should meet the requirements of GB/T191.

7.2 **Packaging**

The enterprise may decide whether toothpaste in pliable tubes shall be packed in little boxes or not, and whether the medium-sized packaging box is made out of contracted packaging or paper box. For large packaging cartons, it should adopt the material that will not damage the materials of the products inside the carton.

7.3 **Transportation**

Loading and unloading should be carried out lightly. Cartons should be piled up according to the arrowheads. Avoid intense vibration, bumping, sunshine, rainfall and damage caused by pressurising.

7.4 **Storage**

7.4.1 Store the products in the ventilated and dry warehouse. Do not place the product close to sources of water or heating systems. Products should be stacked no less than 8cm above the ground or 50cm from the wall. In the middle of warehouse, there should be an aisle. Cartons should be stacked according to the arrowheads, and should not be placed upside down.

7.4.2 **Guarantee period**

7.4.2.1 The guarantee period should be indicated along with the production date shown according to the order of year, month and date, and no reversal of the order is permitted. Years should be indicated by 4-digit figures, e.g. 2000;

7.4.2.2 Limited period of use and production batch;

The guarantee period is fixed by the enterprises, but should not exceed 5 years.

Appendix A

(Standardised Appendix)

Lead content determination of samples directly injected from graphite oven

A1 Instruments and reagents

A1.1   An atomic absorption spectrometer (attached with graphite oven).

Conditions of the machine:

Wavelength: 283.3nm, lamp current 3.5mA, and slit 1.0. Use deuterium light to deduct the background.

The determination conditions of graphite oven are shown in Table A1.

A1

Item	Temperature, ^{o}C	Retention time, s	Temperature rise speed, $^{o}C/s$
Dry	90~120	80	-
Cineration	700	50	-
Atomisation	1,750	2.0	400

A1.2   A 10ml microsyringe.

A1.3   A magnetic force stirrer.

A1.4   Balance: Accuracy at 0.001g.

A1.5   A 50ml straw.

A1.6   Several 100ml beakers.

A1.7   (1+1) nitric acid (of excellent grade and pure).

A1.8   Prepared standard lead solution: weigh 0.1598g of lead nitrate. Use appropriate amount of (1+1) nitric acid solution for dissolving it. Use distilled water to dissolve it, and obtain a predetermined volume to 1,000ml. The concentration of the solution is 100• g/ml.

A1.9 Diluter: take appropriate volume of agar, ammonium dihydrogen phosphate, and dispenser, which are dissolved by distilled water to obtain a certain predetermined volume.

A2 Handling of sample

Take a tube of toothpaste at random as the sample. Weigh 2.00g of toothpaste, and place it in a 100ml beaker. Add 50ml of diluter and 1.0ml (1+1) nitric acid. Stir well on the magnetic force stirrer until an even solution is formed (taking around 20 min.).

A3 Determination of sample

Under the condition that the sample is being stirred on the magnetic force stirrer, use the microsyringe to absorb 10ml (do not absorb the bubble), and fill it into the graphite tube. Turn on the switch of the graphite oven, and carry out determination of

the sample. Record the absorption value, and carry out blank test.

A4 Drawing of standard curve

According to the absorption value of sample, prepare 3~5 standard lead solutions with the corresponding concentration. Determine the absorption value, deduct the blank absorption value, and draw the curves of concentration and absorption value.

A5 Calculation

The sample absorption value is deducted by the blank absorption value. From the standard curve, check the corresponding concentration, which is multiplied by the multiple, resulting in the lead content in the sample toothpaste (mg/kg).

Calculation equation:

$$X = K \bullet c \quad \text{………………………………} \quad (A1)$$

where, X — lead content in toothpaste, mg/kg;

K — diluted multiple of sample;

c — corresponding concentration checked from the standard curve, mg/kg.

A6 Admissible error

The admissible error between the results acquired from both parallel determinations is ± 5%.

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