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## DRAFT EAST AFRICAN STANDARD

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Toothpaste — Specification

EAST AFRICAN COMMUNITY

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## Foreword

Development of the East African Standards has been necessitated by the need for harmonizing requirements governing quality of products and services in the East African Community. It is envisaged that through harmonized standardization, trade barriers that are encountered when goods and services are exchanged within the Community will be removed.

The Community has established an East African Standards Committee (EASC) mandated to develop and issue East African Standards (EAS). The Committee is composed of representatives of the National Standards Bodies in Partner States, together with the representatives from the public and private sector organizations in the community.

East African Standards are developed through Technical Committees that are representative of key stakeholders including government, academia, consumer groups, private sector and other interested parties. Draft East African Standards are circulated to stakeholders through the National Standards Bodies in the Partner States. The comments received are discussed and incorporated before finalization of standards, in accordance with the Principles and procedures for development of East African Standards. XXXXXX.

East African Standards are subject to review, to keep pace with technological advances. Users of the East African Standards are therefore expected to ensure that they always have the latest versions of the standards they are implementing.

The committee responsible for this document is Technical Committee EASC/TC 078, Healthcare and medical devices.

Attention is drawn to the possibility that some of the elements of this document may be subject of patent rights. EAC shall not be held responsible for identifying any or all such patent rights.

This second edition cancels and replaces the first edition (EAS 187:2000), which has been technically revised.

## Introduction

The need for the development of an East African Standard on toothpaste has arisen from the fast expanding toothpaste industry within the region. Toothpaste assists in the removal of daily accumulation of debris and deposits from the exposed surface of the teeth without causing injury to the teeth and mucous membrane of the mouth. In addition it may remove stain and odour but it shall not remove enamel of the teeth.

DRAFT EAST AFRICAN STANDARD

## Toothpaste — Specification

### 1 Scope

This Draft East African Standard specifies the requirements, sampling and test methods for toothpaste (fluoridated and non-fluoridated) for use with a toothbrush in the cleaning of natural teeth.

### 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

EAS 377, *Cosmetics and cosmetic products*

ISO 4833, *Microbiology of the food chain -- Horizontal method for the enumeration of microorganisms*

ISO 6634, *Fruits, vegetables and derived products -- Determination of arsenic content -- Silver diethyldithiocarbamate spectrophotometric method*

ISO 6579-1, *Microbiology of the food chain -- Horizontal method for the detection, enumeration and serotyping of Salmonella -- Part 1: Detection of Salmonella spp*

ISO 21150, *Cosmetics -- Microbiology -- Detection of Escherichia coli*

### 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply. ISO and IEC maintain terminological databases for use in standardization at the following addresses: —ISO Online browsing platform: available at <http://www.iso.org/obp>

#### 3.1

##### **bulk pack**

A pack containing two or more product units of the same nominal volume

#### 3.2

##### **defective**

A product unit that fails to meet one or more of the requirements as set out in 4 and 7.

#### 3.3

##### **dentine**

The calcified tissue, tubular in structure that forms the bulk of a tooth.

#### 3.4

##### **dentifrice**

any substance or combination of substances specially prepared for the public for hygiene of the accessible surfaces of teeth and surrounding tissues



**3.5  
enamel**

highly calcified tissue, prismatic in structure that covers the crown of a tooth.

**3.6  
lot**

Collection of product units of the same size, type and style which have been manufactured under essentially the same conditions

**3.7  
product unit**

One unit of the final product consisting of the closed tube filled with toothpaste. The tube may be contained in a box (usually carton)..

**3.8  
toothpaste**

any semi-solid dentifrice preparation presented in the form of a paste, cream or gel

**3.9  
toothbrush**

device, with the working end of which carries filaments primarily for cleaning surfaces within the oral cavity

## **4 Requirements for toothpaste**

### **4.1 Composition**

4.1.1 The toothpaste shall not contain sucrose or other readily fermentable carbohydrates. Artificial sweeteners, if used, shall comply with the specifications given by FOA/WHO Joint expert committee of food additives.

4.1.2 Colouring matter, if used, shall comply with the requirements of EAS 377-3:

4.1.3 All ingredients shall be of acceptable quality and shall meet the requirements of the EAS 377.

### **4.2 Consistency**

The toothpaste shall be smooth and free from lumps or particles, which are palpable in the mouth as separate or discrete particles.

### **4.3 Homogeneity**

The paste shall extrude from the tube at  $27^{\circ}\text{C} \pm 2^{\circ}\text{C}$  in the form of a homogeneous mass with the application of normal force starting from one end of the tube.

### **4.4 Toxicity**

4.4.1 The toothpaste shall not contain ingredients in sufficient concentration to cause a toxic or irritant reaction when used in the oral cavity, nor shall it be otherwise harmful in normal use.

### **4.5 Stability**

The toothpaste shall not ferment, segregate or otherwise deteriorate, during normal conditions of storage and use. When tested in accordance with annex C, the fore mentioned defects shall not occur.

#### 4.6 Effect of container

4.6.1 Tooth paste shall be packed in suitable tubes of material which shall not corrode, deteriorate, or cause contamination of the tooth paste during normal conditions of storage and use. The paste shall be examined visually by extruding part of the contents. The internal surface of the tube when examined in accordance with Annex D shall show no visible signs of corrosion or deterioration and the toothpaste shall show no sign of contamination.

4.6.2 The lead content of the paints on the tube shall not exceed 250 mg/kg, when tested in accordance with Annex A.

4.6.3 The toothpaste shall extrude to the extent of 94 % of the nominal volume, when tested in accordance with Annex E.

#### 4.7 Fluorine derivatives

The total fluorine derivative and water-soluble fluorine derivative in fluoridated toothpaste (both determined as fluoride ion) shall comply with the requirements given in Table 1. The values given in the table are considered acceptable for toothpaste formulated to contain 1000 mg/kg expressed as fluoride ion. Fluoridated Toothpaste for children, shall be formulated to contain 500 mg/Kg of fluoride expressed as fluoride ion

NOTE — The therapeutic effectiveness of a fluoride toothpaste may be influenced by the combination of fluorine containing additive, abrasive agent and other constituents in toothpaste. Combinations additional to those in the table are acceptable provided that the product complies with all requirements of this standard with specific reference to clause 4. Table 1 — Total and water soluble fluorine derivatives

Predominant abrasive agent in toothpaste	Permitted range of total fluorine derivatives as fluoride ion, mg/kg
Fluoride agent: Stannous fluoride Insoluble sodium metaphosphate Silica Calcium pyrophosphate	Children <sup>a</sup> : 600 to 800 Adults: 900 to 1500
<b>Fluoride agent: Sodium Monofluorophosphate</b> Alumina Calcium carbonate Dicalcium phosphate Insoluble sodium metaphosphate Silica	) ) Children: 600 to 800 ) Adults 900 to 1500 ) )
Fluoride agent: Sodium fluoride High beta phase calcium Pyrophosphate Polymethacrylate spheres	) ) Children: 600 to 800 ) Adults 900 to 1500 ) )
<sup>a</sup> Children shall imply ages 3 to 6 years	

For non-fluoridated toothpaste, Table 1 shall not apply.

Toothpaste with the fluoride level up to 50 mg/kg shall be regarded as non-fluoridated toothpaste.

Test methods specified in Annex F are suitable for sodium monofluorophosphate formulations. If formulations involving other fluoride agents are used, then the test methods have to be approved by the respective partner states National Standards Bodies.

#### 4.8 Other requirements

The toothpaste shall also comply with requirements given in Table 2.

**Table 2 — Requirements for tooth paste**

S/N	Characteristic	Requirements	Method of test
1	Lead (Pb) mg/kg, max	0.5	Annex A
2	pH	5.5 to 10.5	Annex B
3	Fineness	0.1	Annex G
	Particles retained on a 150 micron sieve, per cent by mass, max		
	Particles retained on a 75 micron sieve, per cent by mass, max	2.0	
	Arsenic (As <sub>2</sub> O <sub>3</sub> ) mg/kg, max	2	Annex H

#### 4.9 Microbiological requirements

The toothpaste shall also comply with requirements given in Table 3

**Table 3 — Microbiological requirements for toothpaste**

Characteristic	Requirements	Method of test
Total aerobic bacteria, per gram, max.	100	ISO 4833
Salmonellae per 25 g	Absent	ISO 6579-1
Escherichia coli per 1g grams	Absent	ISO 21150

#### 5 Prohibition or restrictions on clinical claims

The toothpaste shall not be claimed to control the amount of dental plaque, remove calculus, reduce dentine hypersensitivity, reduce the incidence of or treat caries or periodontal disease or any other oral diseases, unless such benefits have been clearly demonstrated by clinical trials or other scientific evidence that are acceptable to the respective partner states National Standards Bodies.

#### 6 Sampling, testing and compliance with the standard.

##### 6.1 Sampling

Sampling shall be done in accordance with annex I

## 6.2 Testing

6.2.1 The toothpaste recovered from the extrusion test shall be well-mixed into a composite sample that will be used for all relevant tests on toothpaste.

6.2.2 A suitable number of empty tubes shall be taken from the sample to test for lead in paint.

## 7 Packing and labelling requirements

### 7.1 Packing

7.1.1 Toothpaste shall be packaged in containers that shall neither show defects nor contaminate the toothpaste during the normal shelf life of the product.

7.1.2 The containers shall be further packed in individual carton boxes or other protective materials.

### 7.2 Labelling

The immediate container (i.e. tube) and outer container shall be legibly and indelibly marked with the following information:

- a) name of the toothpaste or registered trade mark;
- b) the name, in chemical nomenclature, of any ingredient for which special therapeutic properties are claimed, together with the concentration present in the toothpaste;
- c) nominal volume of contents or weight of contents;
- d) code number or batch number;
- e) name and address of manufacturer;
- f) date of manufacturer;
- g) country of manufacture;
- h) in the case of fluoridated toothpaste, the words "FLUORIDE TOOTHPASTE" shall be given in;
- i) expiry date; and
- j) safety notice regarding the use by children below six years of age.

## Annex A (normative)

### Determination of lead content

#### A.1 Principle

The material is brought into solution. The brown colour produced with aqueous hydrogen sulphide solution is matched with that produced with a standard lead solution.

#### A.2 Apparatus

Nessler tubes -100 ml capacity

#### A.3 Reagents

##### A.3.1 Concentrated nitric acid

##### A.3.2 Hydrofluoric acid - 40% (m/m)

##### A.3.3 Citric acid

##### A.3.4 Bromophenol blue indicator

Dissolve 0.1 g of bromophenol blue in 100 ml of rectified spirit.

##### A.3.5 Coppersulphate

##### A.3.6 Hydrogen sulphide gas from Kipp's apparatus

##### A.3.7 Dilute nitric acid - approximately one per cent

##### A.3.8 Ammonium hydroxide

Dilute one volume of liquor ammonia (sp. gr. 0.92) with 10 volumes of water.

##### A.3.9 Thymol blue indicator

Dissolve 0.1 g of thymol blue in 100 ml of rectified spirit.

##### A.3.10 Potassium cyanide solution-10% (m / v).

##### A.3.11 Hydrogen sulphide solution.

Freshly prepared, saturated solution.

##### A.3.12 Standard lead solution

Dissolve 1.600 g of lead nitrate in water and make up the solution to exactly 1 000 ml. Transfer 10 ml of the solution by means of a pipette into a 1000 ml flask and dilute to the mark. One millilitre of the final solution contains 0.01 mg of lead (Pb). The solution should be freshly prepared.

## A.4 Procedure

### A.4.1 preparation of sample

A.4.1.1 Accurately weigh 2.000 g of the toothpaste, in a platinum dish and incinerate for about 2 h at 525 °C to 550 °C. Cool, then add 10 ml of concentrated nitric acid and heat on a steam bath for 2 h.

A.4.1.2 Evaporate off the nitric acid as far as possible, then add 5 ml of water and evaporate to dryness.

A.4.1.3 Take the residue to fumes on a hot plate with three successive portions of hydrofluoric acid. Cool and dilute with water.

A.4.1.4 Filter the solution, if necessary, with suction through a fine fritted glass filter and dilute the filtrate and washings to 200ml in a graduated flask. This solution shall be used in the test in A.4.2 and H.3.

### A.4.2 Method I

A.4.2.1 Take in a beaker 100 ml of the solution prepared in A.4.1. Add 10 g of citric acid and adjust to pH 3.0 to 3.4 by adding ammonium hydroxide (to give a yellow purple colour with bromophenol blue indicator).

A.4.2.2 Add about 10 mg of copper sulphate to act a co-precipitant. Precipitate sulphides by passing hydrogen sulphide until solution is saturated.

A.4.2.3 Dissolve the sulphides, without previous washing, with 5 ml of hot dilute nitric acid, drawing solution through filter into the original flask; wash with hot water and collect the washing along with the solution in nitric acid.

A.4.2.4 Boil to remove sulphureted hydrogen. Add 5 g of citric acid previously dissolved in water, make ammoniacal to pH between 8.5 and 10 (bluish) green to blue towards drop of thymol blue indicator) and add 5 ml of potassium cyanide solution.

A.4.2.5 Transfer to a Nessler tube, add 10 ml of hydrogen sulphide solution, dilute to the mark and shake. Carry out a control test using 2 ml of standard lead solution and the same quantities of other reagents as used in test with the material.

A.4.2.6 The toothpaste shall be taken as not having exceeded the limit prescribed if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

### A.4.3 Alternative method of test for lead contents

#### A.4.3.1 Principle

Lead in toothpaste is extracted with acid after low temperature ashing. The ammonium pyrrolidine dithiocarbamate (APDC) complex is formed, extracted into 4 - methylpentan-2-one and the lead is determined by atomic absorption spectroscopy.

#### A.4.3.2 Precautions

All apparatus and reagents shall be lead free. Avoid contamination, particularly from atmospheric dust. To avoid volatilization of lead it is essential that ashing be carried out at a low temperature 450°C is recommended, 500 °C maximum.

#### A.4.3.3 Apparatus

The following apparatus is required:

A.4.3.3.1 Beakers - 100 ml capacity, borosilicate glass or silica evaporating dishes.

- A.4.3.3.2 Flasks - conical, 100 ml capacity.
- A.4.3.3.3 Funnels - separating 100 ml capacity.
- A.4.3.3.4 Muffle furnace
- A.4.3.3.5 Atomic absorption spectrophotometer and lead hollow cathode lamp
- A.4.3.3.6 Pipette - Pasteur type
- A.4.3.3.7 Pipette 10 ml

#### **A.4.3.4 Reagents**

The following reagents shall be used and all shall be of recognized analytical reagent quality.

##### **A.4.3.4.1 Acetone**

##### **A.4.3.4.2 Ethanol**

##### **A.4.3.4.3 4-methylpentan-2-one (isobutyl methyl ketone)**

##### **A.4.3.4.4 Nitric acid - concentrated**

##### **A.4.3.4.5 Nitric acid - 5M**

##### **A.4.3.4.6 Ammonia solution - 5M solution is recommended but a more dilute solution may be used if preferred**

##### **A.4.3.4.7 APDC one percent solution - Wash about 1.5 g of ammonium pyrrolidine**

dithiocarbamate, (APDC) with 20 ml acetone in a P4 sintered glass crucible. Suck dry, weigh 1 g of the washed APDC and dilute to 100 ml with water.

##### **A.4.3.4.8 Standard lead solution - (1 000 mg/kg lead)**

#### **A.4.3.5 Procedure**

##### **A.4.3.5.1 Destruction of organic matter**

- A.4.3.5.1.1 Accurately weigh about 2 g of the toothpaste into a 100ml beaker or a silica evaporating dish.
- A.4.3.5.1.2 Slurry with 10 ml to 15 ml ethanol and evaporate to dryness on a water bath.
- A.4.3.5.1.3 Place in muffle furnace at 100 °C. Slowly raise the temperature by 50 °C steps to 450 °C to avoid ignition. Complete ashing at 450 °C
- A.4.3.5.1.4 If the ash is dark coloured, wet it with a few drops of concentrated nitric acid and reheat-in the furnace at, 450 °C for 30 s to 1h.

##### **A.4.3.5.2 Acid extraction of the ash**

- A.4.3.5.2.1 Add 5 ml water and 10 ml 5 M nitric acid to the cooled ash and boil gently for 5 min.
- A.4.3.5.2.2 Filter the solution into a 100ml conical flask rinsing with two 5 ml portions of water and retaining as much as possible of any insoluble matter in the beaker or dish.

A.4.3.5.2.3 Add 10 ml 5M nitric acid to the beaker or dish and boil gently for 5 min.

A.4.3.5.2.4 Filter into the same conical flask rinsing with two 5 ml portions of water.

#### A.4.3.5.3 Determination of lead

A.4.3.5.3.1 Adjust the pH value of the acid solution to 3 to 4 with the ammonia solution, transfer to a separating funnel and cool to room temperature.

NOTE The effect of the pH value on extraction of lead is indicated in table A.1

**Table A.1 — The effect of the pH value on extraction of lead**

pH	Peak height (mm)	
	0.5 µg/ml	1.0µg/ml
1	55	100
2	55	122
3	72	142
4	68	136
5	66	108
6	54	115
7	44	92
8	50	90

A.4.3.5.3.2 Add 2 ml of APDC solution, mix and set aside for 5 min.

A.4.3.5.3.3 Add 10 ml of 4 methylpentan-2-one, measured accurately using pipette, and shake vigorously for 1 min.

A.4.3.5.3.4 Allow to separate, remove the clear organic layer with a Pasteur pipette.

A.4.3.5.3.5 Aspirate the organic layer into the atomic absorption spectrophotometer fitted with a lead hollow cathode lamp set at 283.3 N/m.

#### A.4.3.6 Calibration

Submit aliquot portions of standard lead solution containing, 0 to 20 µg Pb to the procedure in A.4.3.5.3. Construct a new curve for each set of measurements.

#### A.4.3.7 Calculation

Relate the test result to the calibration curve and calculate, µg Pb/9 (mg/kg).



**Annex B**  
**(normative )**  
**Determination of pH**

**B.1 Apparatus**

**B.1.1 Analytical balance**

**B.1.2 pH meter with appropriate glass/calomel electrode assembly**

**B.1.3 Stirrer**

**B.2 Procedure**

B.2.1 Weigh  $5.0 \text{ g} \pm 0.5 \text{ g}$  of toothpaste into a 100 ml beaker. Slurry with  $20 \text{ ml} \pm 0.1 \text{ ml}$  of distilled water.

B.2.2 Determine the pH value of the slurry at a temperature of  $23 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$  using the glass/calomel electrode assembly, about 3 min after the commencement of slurring.

**B.3 Expression of results**

Report the pH value of the toothpaste.

**Annex C**  
**(normative)**  
**Test for stability**

**C.1 Apparatus**

Oven

**C.2 Procedure**

C.2.1 Transfer a suitable quantity of the composite sample of the tooth paste into each of two glass test tubes and stopper them.

C.2.2 Heat the two tubes for 72 hours in an oven maintained at  $45\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ . Allow to cool and examine the contents visually for homogeneity and for signs of fermentation or other deterioration or both.

**C.3 Expression of results**

Report the result as pass or fail

## Annex D

(normative)

### Determination of corrosion of the container

#### D.1 Procedure

D.1.1 Heat the tube of toothpaste at  $45\text{ °C} \pm 1\text{ °C}$  for ten days and then test as follows:

D.1.2 Extrude a portion of the paste and examine it for visible contamination. Slit the tube, remove the paste and examine the surfaces of the tube for signs of corrosion, chemical attack or other damage.

D.1.3 Disregard surface staining by colouring agents in the toothpaste.

#### D.2 Expression of result

Report the result as pass or fail

## Annex E (normative)

### Determination of extrusion

#### E.1 Procedure

E.1.1 Maintain the tube of toothpaste at  $23\text{ °C} \pm 2\text{ °C}$  for at least 2 hours and then test as follows: Remove the cap and invert the tube over a plastics beaker for which a tare has been recorded.

E.1.2 Press-out the paste from the fold of the tube in the direction of the shoulder, holding the tube between thumb and index finger of the left and right hand.

E.1.3 When the tube is almost emptied, the mantle of the tube is to be pulled over an edge, removing the rest of the paste in the shoulder by nipping it off rectangular. The extrusion shall be carried out without the application of abnormal force.

#### E.2 Calculation and expression of results

E.2.1 Record the mass,  $m$ , of toothpaste extruded. Use a suitable method to determine the density of the toothpaste and hence calculate the volume of toothpaste extruded.

E.2.2 The extrusion, expressed as % (V/V) of the nominal volume, is given by the formula

$$\frac{m}{\rho \times \text{nominal volume}} \times 100$$

Where

$m$  is the mass of toothpaste extruded;

$\rho$  is the density of the toothpaste.

## Annex F (normative)

### Determination of fluoride content

#### F.1 Principle

The fluorine derivative species in a sample of toothpaste (for total fluoride analysis) or in the aqueous extract of the toothpaste (for soluble fluoride analysis) is allowed to react with trimethylchlorosilane under acidic conditions to produce trimethylfluorosilane. This derivative is extracted into toluene containing n-pentane as internal standard and the organic phase is analysed by gas chromatography.

#### F.2 Reagents

Use only analytical reagent grade and distilled or demineralized water.

##### F.2.1 Hydrochloric acid, concentrated.

##### F.2.2 n-Pentane solution in toluene (soln A).

0.6 g of n-pentane made up to 1 litre with toluene.

##### F.2.3 Standard fluoride solution (soln B).

An aqueous solution of sodium fluoride containing 1.0 mg F/mL.

##### F.2.4 Trimethylchlorosilane.

#### F.3 Apparatus

##### F.3.1 Gas chromatograph

A suitable gas chromatograph equipped with a flame ionization detector, and having a column of length about 1.5 m and nominal outside diameter 6 mm packed with 10% methyl silicone (100%) on 80-100 mesh flux calcined diatomite, acid washed. The recommended GC instrument conditions are as follows:

Temperatures - Column 50 °C

Injector 80 °C

Detector 100 °C

Carrier gas - Nitrogen, flow rate 20 ml/min

### F.3.2 Centrifuge

### F.3.3 Microsyringe, 1 $\mu$ l

## F.4 Determination of response factor of the apparatus

**F.4.1** Pipette 1 ml of the fluoride solution into a 50 ml plastics flask. Using a measuring cylinder, add 8 ml of concentrated hydrochloric acid, stopper the flask and mix the contents. Add from a burette 2.0 ml of the trimethylchlorosilane.

**F.4.2** Securely stopper the flask, mix well by shaking and allow to stand for 15 min to cool. Using a safety pipette, pipette 5 ml of the toluene solution of n-pentane into the flask and stopper. Mix thoroughly by shaking vigorously for 2 min.

**F.4.3** Transfer the contents of the flask to a centrifuge tube, stopper and centrifuge at 2500 r/min for 5 min. Inject 0.6  $\mu$  l of the upper layer onto the column and allow the chromatogram to develop.

**F.4.4** Draw tangent base lines to the peaks of interest, and measure the heights of the trimethylfluorosilane and n-pentane peaks. ((Alternatively, determine the areas of the relevant peaks).

The response factor (k) is given by the formula

$$\frac{\text{Peak height of trimethylfluorosilane}}{\text{Peak height of n - Pentane}} \times \frac{\text{grams of n - Pentane in 5 ml of solution A}}{\text{grams of fluoride in 1 ml of solution B}}$$

## F.5 Procedure

### F.5.1 For soluble fluoride

**F.5.1.1** Weigh accurately  $5.0 \pm 1.0$  of toothpaste into a centrifuge tube. Add approximately 15 ml of water, slurry with a glass rod and centrifuge for 10 min. Decant the supernatant liquid into a 50 ml volumetric flask. Repeat this extraction process an additional two times and bring the volume of the supernatant liquid up to 50 ml with water.

**F.5.1.2** Pipette 10 ml of the above solution into a plastics centrifuge tube. Add 8 ml of concentrated hydrochloric acid and immerse in a boiling water bath for 1 min and let stand for 10 min. Then cool under running water.

**F.5.1.3** Add 1 ml of trimethylchlorosilane, cap, mix and let stand for 15 min. Pipette 5 ml of n-pentane solution into the tube, mix centrifuge and inject 0.6  $\mu$ L of the upper layer.

**F.5.1.4** The mass of fluoride, in grams, is given by the formula

$$\frac{\text{Peak height of trimethylfluorosilane}}{\text{Peak height of n - Pentane}} \times \frac{\text{grams of n - Pentane in 5 ml of solution A}}{k}$$

### F.5.2 For total fluoride

**F.5.2.1** Weigh accurately  $1.0 \pm 0.1$  g of toothpaste into a plastics centrifuge tube. Add several glass beads and 8 ml of concentrated hydrochloric acid in parts. Cap and shake, pausing several times to release pressure.

**F.5.2.2** Add 8 ml of water, cap and mix. Immerse in a boiling water bath for 1 min. Mix and let stand for 2 min, then cool under running water.

F.5.2.3 Add 1 ml of trimethylchlorosilane, mix and let stand for 15 min. Pipette 5 ml of n-pentane solution into the tube, mix, centrifuge and inject 0.5 µl of the upper layer.

The mass of fluoride, in grams, is given by the formula

$$\frac{\text{Peak height of trimethylchlorosilane}}{\text{Peak height of n - Pentane}} \times \frac{\text{grams of n - Pentane in 5 ml of solution A}}{\text{grams of fluoride in 1 ml of solution B}}$$

### F.5.2.3 Expression of result

Report the results of total and soluble fluorine derivative of the sample tested as mg/kg of fluoride ion.

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## Annex G (normative)

### Determination of fineness

#### G.1 Procedure

##### G.1.1 Equipment

On 150-micron sieve

##### G.1.2 Determination

D.1.2.1 Place about 10 g of the toothpaste, accurately weighed, in a 100 ml beaker. Add 50 ml of water and allow to stand for 30 min. with occasional stirring until the tooth-paste is completely dispersed.

D.1.2.3 Transfer to a 150-micron sieve and wash by means of a slow stream of running tap water and finally with a fine stream from a wash bottle until all the matter that can pass through the sieve has passed.

D.1.2.4 Let the water drain from the sieve and then dry the sieve containing the residue in an oven. If there is any residue on the sieve, carefully transfer it to a tared watch glass and dry it to constant mass in an oven at  $105\text{ °C} \pm 2\text{ °C}$ .

##### G.1.3 Calculation

Material retained on 150-micron

$$\% \text{ by mass} = \frac{M_1 \times 100}{M_2}$$

Where

$M_1$  is the mass in g of residue retained on the sieve;

$M_2$  is the mass in g of the material taken for the test.

#### G.2 Procedure II

##### G.2.1 Equipment

On 75-micron sieve

##### G.2.2 Determination

Weigh accurately about 10 g of the toothpaste and proceed as in G.1.2, using a 75-micron sieve. If there is any residue on the sieve carefully transfer it to a tared watch glass and dry it to constant mass in an oven at  $105\text{ °C} \pm 2\text{ °C}$ .

##### G.2.3 Calculation

Calculate as in G.1.3.



## Annex H (normative)

### Determination of arsenic

#### H.1 modified Gutzeit method

##### H.1.1 Outline of the method

H.1.1.1 The modified Gutzeit method of test of Arsenic shall be employed in cases where arsenic content is not needed and only a knowledge of is desired. In cases where the actual arsenic content is to be determined, silver diethyldithiocarbamate method outlined in ISO 6634 shall be followed.

H.1.1.2 Arsenic present in a solution of the material is reduced to arsine which is made to react with mercuric bromide paper. The stain produced is compared with a standard stain.

##### H.1.2 Reagents

###### H.1.2.1 Mixed acid

Dilute one volume of concentrated sulphuric acid with four volumes of water, and add to this 10 g of sodium chloride for each 100 ml of the solution.

###### H.1.2.2 Ferric ammonium sulphate solution

Dissolve 84 g of ferric ammonium sulphate in water containing 10 ml of mixed acid and make up to one litre.

###### H.1.2.3 Concentrated hydrochloric acid

###### H.1.2.4 Stannous chloride solution

Dissolve 80 g of stannous chloride ( $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ ) in 100 ml of water containing 5 ml of concentrated hydrochloric acid.

##### H.1.3 Procedure

H.1.3.1 Place 2g of toothpaste sample accurately weighed in a platinum dish and incinerate for about 2 hours at 525°C to 550°C. Cool and add 1ml to 2 ml of hydrochloric acid and 0.5 ml nitric acid and evaporate to dryness on the steam bath.

H.1.3.2 Dissolve the residue in 5 ml hot water, evaporate to dryness and treat it with hydrofluoric acid. Evaporate again to dryness.

H.1.3.3 Dilute it with water (about 50 ml). Filter the solution, if necessary, with suction through a fine fritted glass filter and dilute the filtrate and washing to 100 ml in a graduated flask.

H.1.3.4 Carry out the test by adding into the Gutzeit bottle 2 ml of ferric ammonium sulphate solution, 0.5 ml of stannous chloride solution and 50 ml of the solution as prepared in A.4.1. For comparison, prepare a stain using 0.001 mg of arsenic trioxide.

## Annex I (normative)

### Sampling

#### I.1 General requirements of sampling

In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

- I.1.1 Samples shall be taken in a protected place not exposed to damp air, dust or Soot.
- I.1.2 The sampling instrument shall be clean and dry.
- I.1.3 The samples, the material being sampled, the sampling instrument and the containers for samples shall be protected from adventitious contamination.
- I.1.4 The samples shall be placed in clean and dry glass containers. The sample containers shall be of a size such that they are almost completely filled by the sample.
- I.1.5 Each container shall be sealed air-tight after filling and marked with full details of sampling, batch or code number, name of manufacturer, and other important particulars of the consignment.
- I.1.6 The samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature and they are protected from light.
- I.1.7 Sampling shall be done by a person agreed to between the purchaser and the supplier, and in the presence of the purchaser or his representative and the supplier or his representatives.

#### I.2 Sampling of packages

- I.2.1 General - The sampling procedure for packages shall consist essentially in selecting and drawing, a sufficient number of unit packs.
- I.2.2 Lot - In a single consignment, all the packages containing toothpaste of the same type and form, representing the same batch of manufacture, shall constitute a lot. If the consignment consists of packages containing toothpaste of different types or forms or batches of manufacture, then the packages containing products of the same type, form and batch of manufacture shall be grouped together; each group shall constitute a separate lot.
- I.2.3 Scale of Sampling -For ascertaining the conformity of a lot to the requirements prescribed in the specifications for individual toothpaste and toilet goods, tests shall be carried out on each lot separately. The number ( n ) of packages to be selected for drawing the samples shall depend on the size ( N ) of the lot in accordance with Table I.1.

Table I.1 — Scale of sampling for packages

No. of packages in the lot(N)	No. of packages to be selected,(n)
Upto 3	Each container
4 to 50	3
51 to 150	4
151 to 300	5
301 to 500	6
501 and above	7

I.2.4 The packages shall be selected at random and to ensure randomness of selection, random number tables shall be used. In case such tables are not available, the following procedure may be adopted: 'Starting from any package, count all the packages in one order as 1,2,3 . . . . . up to r and so on, where r is the integral part of  $N/n$ . Every  $r^{\text{th}}$  package thus counted shall be withdrawn to give a sample for purposes of test '.

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