IS 6356 (2001): Toothpaste [PCD 19: Cosmetics]

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भारतीय मानक

टूथपेस्ट — विशिष्टि

(तीसरा पुनरीक्षण)

Indian Standard

TOOTHPASTE — SPECIFICATION

(Third Revision)

ICS 11.060.10

BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Price Group 8

Cosmetics Sectional Committee, PCD 19

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Cosmetics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was originally issued in 1971 and first revised in 1978 when the changes were made in the requirements of fineness and a test for hard and sharp edged particles was included in place of test for abrasion.

In the second revision in 1993, toothpaste was classified as fluoridated and non-fluoridated toothpaste. New requirements for toothpaste stability, microbial purity, spreadability, ease of extrusion, fluoride ion for fluoridated toothpaste and tube inertness were included. Lower limit for *p*H was raised in line with International standard, keeping in mind the safety aspect of toothpaste. Requirements for expiry date and labelling key ingredients on containers were also incorporated in this revised version.

In this version cognizance has been taken of new and emerging technologies in the field of oral care and dentistry. Extensive help has been taken from various overseas standards for toothpastes, as listed below:

AS 2827 : 1982 Toothpaste — Specification
SABS 1302 : 1980 Toothpaste — Specification
ISO 11609 : 1995 (E) Dentistry — Toothpaste requirements, Test methods and marking
BS 5136 : 1981 Specification for toothpaste
SLS 275 : 1980 Specification for toothpaste
BDS 1216 : 1989 Specification for toothpaste

An attempt has been made to incorporate relevant parts of these standards while keeping in mind specific needs in Indian context.

The main theme of safety of the consumer while using the product is retained as central in this version. Certain new tests have been incorporated while certain existing tests have been modified/standardized in the light of the available knowledge. The salient features of this revision are as follows:

The toothpaste, when used in a normal manner, shall not cause injury to the teeth, gums, and mucous membrane of the mouth or the body in general. The role of toothpaste is to clean the surface of teeth and also to prevent / reduce the incidence of oral dental diseases like caries, gingivitis or periodontal diseases. The use of toothpaste improves the oral hygiene. Hence toothpaste with active ingredients like fluoride, triclosan, etc, which help in improving oral hygiene are part of this specification.

A definition for dentifrice in general and toothpaste in particular has been included from International Standard.

In line with the new emerging technologies such as use of soft agglomerates/capsules, the fineness specifications and test method has been modified in this version.

Toothpaste formulations are designed to have optimal abrasivity to effect cleaning action without hurting soft tissues or tooth enamel by way of excessive abrasivity. Relative dentine abrasivity (RDA) is recognized in most of the International Standards as the measure to determine this parameter. RDA is, therefore, recommended as a type test in this standard. The abrasivity of the toothpaste shall not exceed the limits specified when tested as per procedure given in Annex H. The abrasivity measurement methodology has been based on ISO 11609 : 1995 (E). Since RDA measurement facility is currently not available in India , a simpler abrasion test using a photographic paper is being developed. This procedure may be adopted in future after completing the studies and establishing validation with RDA.

Toothpaste containing fluorides have been unequivocally proven to be effective in caries control but under

(Continued on third cover)

Indian Standard

TOOTH PASTE —SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for toothpaste.

2 REFERENCES

The Indian Standards referred in this standard have been listed below. Other standards which are necessary adjuncts to this standard are given in Annex J. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standard:

IS No.	*Title*
264 : 1976	Nitric acid (*second revision*)
265 : 1993	Hydrochloric acid (*fourth revision*)
460 (Part 1) : 1985	Test sieves: Part 1 Wire cloth test sieves (*third revision*)
2088 : 1983	Methods for determination of arsenic (*second revision*)
3958 : 1984	Methods of sampling cosmetics (*first revision*)
4707	Classification of cosmetic raw materials and adjuncts
(Part 1) : 2001	Dyes colours and pigments (*second revision*)
(Part 2) : 2001	List of raw materials generally not recognized as safe for use in cosmetics (*second revision*)
14648 : 1999	Methods of test for microbiological examinations of cosmetics

3 DEFINITIONS

3.1 Dentifrice

A dentifrice is any substance or combination of substances specially prepared for the public for cleaning the accessible surfaces of teeth.

3.2 Toothpaste

A toothpaste is defined as a dentifrice in the form of a smooth, semisolid, homogeneous mass containing acceptable ingredients such as abrasives/polishing agents, surface active agents, humectants, binding agent, and other appropriate substances for oral health maintenance. The product can be opaque, transparent, or combination thereof, coloured or white, packed in a suitable container from which it can be extruded in the form of a continuous mass.

4 TYPES

The toothpaste shall be of either Type 1 or Type 2:

Type 1 — Non-Fluoridated

Type 2 — Fluoridated

5 REQUIREMENTS

5.1 Composition

A toothpaste shall not contain mono or disaccharides, for example, sucrose or other readily fermentable carbohydrates. All the raw materials used shall conform to respective Indian Standards wherever they exist. A list of ingredients conventionally used in the manufacture of tooth paste is given in Annex A for information only.

The dyes and pigments used in manufacture of toothpaste shall comply with the provisions of IS 4707 (Part 1).

Ingredients other than dyes and pigments used in formulation of toothpaste shall comply with the provisions of IS 4707 (Part 2).

5.2 Dispensing

The paste shall extrude from the collapsible tube or any other suitable container in which it is packed, at 27 ± 2°C in the form of continuous mass with the application of normal force, without the application of excessive force which would cause injury to the tube or the container. It shall be possible to extrude bulk of the contents from the container or the tube starting from the crimped end of the tube by rolling the tube gradually.

5.3 Stability

The toothpaste shall not show any physical sign of deterioration during normal conditions of storage and use. When subjected to a temperature of 45 ± 2°C for a period of 28 days the toothpaste shall meet the requirements of the standard. When cooled to a temperature of 5°C for 1 hour, after taking out and pressing tube, the paste shall be found extrudable from the tube and meet the requirement of this standard.

5.4 Packaging Material Inertness

The collapsible tubes or any other suitable container used for packaging of toothpaste shall not corrode, deteriorate or cause contamination of the toothpaste during normal condition of storage and use. When subjected to a temperature of 45 ± 2°C for 10 days, the paste shall then be examined visually by extruding part of the contents. The internal surface of the tube shall be examined after slitting it open and removing the remaining contents. There should be no sign of corrosion, chemical attack or other damage.

5.5 Acceptance Test

The toothpaste shall also comply with the requirements given in Table 1 when tested according to the methods given in Annex B to Annex G, as per reference made in col 5 of Table 1.

5.6 Shelf Life

Shelf life shall be declared by the manufacturer for all types of toothpaste.

Manufacturing date (month and year) should be mentioned on tube and carton. The expiry date or 'Best use before' shall be mentioned on the tube and carton. During the shelf life the product will meet the requirement of the standard.

5.7 Additional Requirements for ECO-Mark (Optional)

5.7.1 *General Requirements*

5.7.1.1 The product shall conform to the requirements for quality, safety and performance prescribed under **5.7.1.2** to **5.7.1.5**.

5.7.1.2 All the ingredients that go into formulation of cosmetics shall comply with the provisions of IS 4707 (Part 1) and IS 4707 (Part 2). The product shall also meet specific requirements as given in the standard.

5.7.1.3 The product package shall display a list of key ingredients in descending order of quantity present.

5.7.1.4 The product shall not be manufactured from any carcinogenic ingredients.

5.7.1.5 The manufacturer shall produce to BIS environmental consent clearance from the concerned State Pollution Control Board as per the provisions of the *Water (Prevention and Control of Pollution) Cess Act,* 1977 and the *Air (Prevention and Control Pollution) Act*, 1981 along with the authorization, if required under the *Environment (Protection) Act*, 1986 and the Rules made thereunder, while applying for ECO-Mark. Additionally, provisions of the *Drugs and Cosmetics Act*, 1940 and the Rules thereunder shall also be complied with.

5.7.2 *Specific Requirements*

Heavy metals calculated as lead (Pb) and arsenic (As_2O_3) shall not exceed 20 and 2 ppm, respectively when tested by the respective method prescribed in Indian Standards.

6 ABRASIVITY (TYPE TEST)

The toothpaste shall not exceed the limits of dentin

Table 1 Requirements for Toothpaste

(*Clause* 5.5)

Sl No.	Characteristic	Requirement for		Method of Test, Ref to Annex
		Non-fluoridated	Fluoridated	
(1)	(2)	(3)	(4)	(5)
i)	Fineness:			
	a) Particles retained on 150 micron IS Sieve, percent by mass, *Max*	10	1.0	B
	b) Particles retained on 75 micron IS Sieve, percent by mass, *Max*	2.5	2.5	—
ii)	*p*H of aqueous suspension	5.5 to 10.5	5.5 to 10.5	C
iii)	Heavy metals (as lead), parts per million, *Max*	20	20	D
iv)	Arsenic (as As_2O_3), parts per million, *Max*	2	2	E
v)	Foaming power, ml, *Min*[1)]	50	50	F
vi)	Available Fluoride ion, parts per million, *Max*	50	1 000	G
vii)	Microbial counts:			
	a) Total viable counts per gram, *Max*	1 000	1 000	IS 14648
	b) Gram negative pathogens per gram, *Max*	Absent	Absent	IS 14648

NOTE — If all the raw materials used in the toothpaste formulation have been tested for heavy metals and arsenic and comply with the requirement, then manufacturer may not test the finished cosmetic for heavy metals and arsenic.

[1)] Applicable to foaming toothpaste only.

abrasivity that of 2.5 times when tested as per the procedure given in Annex H.

It may be noted that type test is recommended to be done on the formulation only once to pass the above criterion. This test need not be done for each and every batch. However, the type test is a must again if the abrasive system is changed in the formulation. It is not required for the new formulation as long as abrasive components are not changed.

7 PACKING AND MARKING

7.1 Packing

Toothpaste shall be packed in collapsible tubes or in any other suitable containers like sachets, pumps or other suitable dispensing systems. When packed in containers, the containers shall be properly sealed and have a leak-proof cap or closure. The containers, if necessary, may further be packed in cartons or any other suitable packaging material.

7.1.1 The material for product packaging shall meet the parameters evolved under the scheme of labelling environment friendly packaging/packaging materials.

7.2 Marking

The tubes and the cartons shall be legibly marked with the following information:

a) Name and type of toothpaste;
b) Name and address of the manufacturer;
c) Net mass or volume of the material in the tube;
d) Batch number, in code or otherwise;
e) Month and year of manufacture;
f) Fluoride ion content in ppm for Type 2 toothpaste;
g) Expiry date or 'Best use before. . . .' (month and year to be declared by the manufacturer);

NOTE — This requirement is exempted in case of pack sizes of 10g/25 ml or less and if the shelf life of the product is more than 24 months.

h) Foaming/non-foaming; and
j) List of key ingredients.

NOTE — This is exempted in case of pack sizes of 30g/60 ml or less.

7.2.1 *BIS Certification Marking*

The containers may also be marked with the Standard Mark.

7.2.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

7.2.1.2 If the product is covered under ECO-Mark (optional), it shall be suitably marked with ECO-Mark logo besides Standard Mark. The label may clearly specify that ECO-Mark is applicable to the contents or the package or both, as the case may be. If the product package is not separately covered under ECO-Mark scheme, it shall be clearly mentioned on the product that ECO-Mark label is applicable to contents only.

8 SAMPLING

8.1 Representative test samples of the material shall be drawn as prescribed in IS 3958.

8.2 Number of Tests and Criteria for Conformity

The tests for abrasivity, stability and container's inertness shall be type tests and shall be performed for product approval whereas tests for dispensing, fineness, *p*H, heavy metals, arsenic, foaming power, fluoride content and microbial counts shall be carried out on each batch for acceptance of the product.

8.2.1 The type tests shall be repeated in the event of change in the basic formulation or whenever there is a change in the type of container being used. However, the acceptance tests shall be performed on each and every batch.

8.3 A batch may be defined as consisting of any quantity of toothpaste manufactured in a single mix at one time.

8.4 The lot shall be declared as conforming to requirements of the specification, if all the test results on each individual sample meet the requirements prescribed in **5.2** to **5.6**.

9 QUALITY OF REAGENT

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities, which affect the results of analysis.

ANNEX A

(*Clause* 5.1)

INGREDIENTS CONVENTIONALLY USED IN THE MANUFACTURE OF TOOTHPASTE

A-1 Raw material used in toothpaste formulation falls into the following categories:

1. Polishing agents
2. Surface active agents
3. Humectant
4. Binding agent
5. Others as per IS 4707 (Part 1) and IS 4707 (Part 2)

A-1.1 Polishing Agents

The principal polishing agents generally used are one or more of those given below:

1. Calcium carbonate
2. Magnesium carbonate
3. Dicalcium phosphate
4. Insoluble sodium metaphosphate
5. Hydrated alumina
6. Aluminium hydroxide
7. Aluminium/Alumino silicate
8. Alumina
9. Alumina fumed
10. Calcium phosphate
11. Calcium pyrophosphate
12. Dicalcium phosphate dihydrate
13. Kaolin
14. Magnesium silicate
15. Potassium metaphosphate
16. Silica gel or precipitated
17. Silica fumed
18. Silica hydrated
19. Sodium aluminium silicate
20. Sodium bicarbonate
21. Sodium metaphosphate
22. Hydroxyapatite
23. Zirconium silicate
24. Sodium polymetaphosphate
25. Tungsten carbide
26. Pumice
27. Silicon carbide
28. Magnesium silicate
29. Agglomerates (mainly consisting of one or more of the polishing agents)
30. Others as per IS 4707 (Part 1) and IS 4707 (Part 2)

A-1.2 Surface Active Agents

The surface active agent(s) generally used are one or more of those given below:

1. Soap
2. Sodium ricinoleate
3. Sodium sulphoricinoleate
4. Sodium lauryl sulphate
5. Sodium alkyl sulphoacetate
6. Sodium salt of sulphated monoglyceride
7. Sodium lauryl sarcosinate
8. Sodium alpha olefin sulphonate
9. Coco-amido-propyl-betaine
10. Sodium dodecyl benzene sulphonate
11. Sodium lauryl sulphoacetate
12. Coconut monoglyceride sulphonates
13. Dioctylsodium-sulphosuccinate
14. Magnesium lauryl sulphate
15. Sodium alkyl benzene sulphonate
16. Sodium alkyl sulphate
17. Sodium lauryl ether sulphate
18. Coco-betaine
19. Others as per IS 4707 (Part 1) and IS 4707 (Part 2)

A-1.3 Humectants

The humectants generally used are one or more of those given below:

1. Glycerol
2. Sorbitol
3. Maltitol
4. Mannitol
5. Polyethylene glycol
6. Propylene glycol
7. Lactitol
8. Xylitol
9. Others as per IS 4707 (Part 1) and IS 4707 (Part 2)

A-1.4 Binding Agents

The binding agents generally used are one or more of those given below:

1. Gum tragacanth
2. Gum karaya
3. Sodium alginate

4. Sodium carboxymethyl cellulose
5. Gelatine
6. Guar gum and its derivatives
7. Xanthan gum
8. Carraghenates
9. Carboxy methyl cellulose
10. Magnesium aluminium silicate
11. Starch
12 Hydroxy propyl cellulose
13. Hydroxy ethyl cellulose
14. Carbopol
15. viscarin
16. Polymers (propylene oxide-ethylene oxide-block copolymer)
17. Others as per IS 4707 (Part 1) and IS 4707 (Part 2)

A-1.5 Other Substances

1. Fluorides of sodium and stannous
2. Monofluorophosphate of ammonium, potassium and sodium
3. Sweeteners, saccharine sodium, aspartarme
4. Petroleum jelly-silicon defoaming compounds and mineral oil
5. Colouring agents
6. Essential oils
7. Flavouring agents
8. Astringents
9. Preservatives
10. Antibacterial agents
11. Antiplaque agents
12. Antitartar agents
13. Whitening agents
14. Anti caries agents
15. Calcium glycerophosphate
16. Granules/agglomerates
17. Anti gingival agents
18. Other as per IS 4707 (Part 1) and IS 4707 (Part 2)

ANNEX B

[*Clause* 5.5 and *Table* 1, Sl No. (i)(a)]

DETERMINATION OF FINENESS

B-1 OUTLINE OF THE METHOD

Squeeze the toothpaste and feel the presence of the particles/agglomerates/granules. Subject the toothpaste suspension to an ultrasonoic treatment and pass through fineness test. Ultrasonification loosens out the soft agglomerates into the constituent materials.

B-2 APPARATUS

B-2.1 Ultrasonic Bath — Trans-O-Sonic Compact model or equivalent,

(60 ± 10 Watts power with 35 ± 5 KHz Ultrasonic frequency, 1-2 Watts /inch 2 power density, L × B × H : 225 × 125 × 60 mm tank).

B-2.2 Sieves — 75 and 150-microns [conforming to IS 460 (Part 1)].

B-2.3 Glass beakers (250 ml, 500 ml) and stirring rods.

B-3 PROCEDURE

B-3.1 Determination of Particle Feel on Butter Paper

Extrude the paste about 15 to 20 cm length each from at least ten collapsible tubes on a butter paper. Test the paste by pressing it along its entire length by a finger for the presence of particles. The toothpaste suspension should be subjected to an ultrasonic treatment followed by a fineness test as described in **B-3.2** and **B-3.3**.

B-3.2 Determination of Particle Size on 150-Micron IS Sieve

Place about 20g of the toothpaste, accurately weighed, in a 250-ml beaker. Add 200 ml of water and allow to stand for about 30 minutes with occasional stirring until the toothpaste is completely dispersed free of toothpaste/gel flocks trapping the agglomerates. Transfer the beaker in an ultrasonic bath. Fill the

Ultrasonic bath (2 litre capacity) to about three-fourth height with water. Clamp the above beaker in the centre of the bath keeping about 1 cm clearance from the bottom of the bath and subject ultrasonification for 10 min to completely loosen out the constituents.

Transfer this suspension quantitatively to a 150 micron IS 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. Let the water drain out and then dry the sieve containing the residue in an oven. If there is any residue on the sieve, carefully transfer it to a tarred watch glass and dry it to constant mass in an oven at 105 ± 2°C.

B-3.3 Determination of Particle Size on 75-Micron IS Sieve

Weigh accurately about 20 g of the toothpaste and proceed as in **B-3.2**, using a 75-micron IS 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 ± 2°C.

B-4 CALCULATION

Material retained on 150-micron IS Sieve, percent by mass $= \frac{M_1 \times 100}{M}$

where

M_1 = mass in g of residue retained on the sieve, and

M = mass in g of the material taken for the test.

ANNEX C

[*Clause* 5.5 and *Table* 1, *Sl No*. (ii)]

DETERMINATION OF *p*H

C-1 PROCEDURE

Dispense 10 g of the toothpaste from the container in a 50-ml beaker and add 10 ml of freshly boiled and cooled water (at 27°C) to make 50 percent aqueous suspension. Stir well to make a thorough suspension. Determine the *p*H of the suspension within 5 min, using a *p*H meter.

ANNEX D

[*Clause* 5.5 and *Table* 1, *Sl No.* (iii)]

DETERMINATION OF HEAVY METALS

D-1 OUTLINE OF THE METHOD

The colour produced with thioacetamide reagent in test solution is matched against that obtained with standard lead solution.

D-2 APPARATUS

D-2.1 Nessler Cylinders — 50 ml capacity.

D-2.2 Weighing Scale — 0.000 1 g accuracy.

D-2.3 Volumetric Flasks — 1 00 ml capacity.

D-2.4 Platinum Crucible

D-2.5 Pipette — 2, 10 ml.

D-3 REAGENTS

D-3.1 Concentrated Hydrochloric Acid — *See* IS 265.

D-3.2 Concentrated Nitric Acid — *See* IS 264.

D-3.3 Hydrofluoric Acid

D-3.4 Dilute Acetic Acid — 6 M (342 ml of glacial acetic acid diluted to 1 000 ml with water).

D-3.5 Glycerol Mixture — Take 15 ml of 1 M NaOH and add 5 ml water and 20 ml of 85 percent glycerol. Mix well.

D-3.6 Thioacetamide Reagent — Weigh 80 mg of thioacetamide and add 2 ml water to it. Shake to dissolve. Add 10 ml glycerol mixture, heat on water bath for 20 s, cool and use immediately.

D-3.7 Lead Nitrate Stock Solution — (100 ppm as Pb) — Dissolve 0.159 9 g of lead nitrate in water containing 1 ml of nitric acid and make up the solution to 1 000 ml.

D-3.8 Standard Lead Solution — (10 ppm as Pb) — Dilute 10 ml of lead nitrate stock solution with water to 100 ml. Each ml is equivalent to 0.01 mg of Pb.

D-3.9 Acetate Buffer (3.5 *p*H) — Dissolve 25 g of ammonium acetate in 25 ml water and add 38 ml of 7M hydrochloric acid. Adjust the *p*H to 3.5 either 2M hydrochloric acid or 6M ammonia and dilute to 100 ml with water.

D-4 PROCEDURE

D-4.1 Place 2g of toothpaste sample accurately weighed in a platinum dish and incinerate for about 2 hours at 525 to 550°C. Cool and add 1 to 2 ml of hydrochloric acid and 0.5 ml nitric acid and evaporate to dryness on the steam bath. Dissolve the residue in 5 ml hot water, evaporate to dryness and treat it with hydrofluoric acid. Evaporate again to dryness. 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. This solution shall be used for tests given in **D-4.2** and **E-3** as test solution.

D-4.2 Transfer 25 ml of test solution prepared in **D-4.1** in a 50 ml Nessler cylinder, add further 2 ml of test solution and 2 ml acetate buffer (*p*H = 3.5) and mix well. Add 1.2 ml of thioacetamide reagent, mix, and immediately dilute with water to 50 ml and allow to stand for 2 minutes.

D-4.3 In the second Nessler cylinder, place 1 ml standard lead solution (*see* **D-3.8**) and add 2 ml of test solution. Dilute with water to 25 ml, and add 2 ml Acetate buffer (*p*H 3.5). Mix, add 1.2 ml of thioacetamide reagent, and immediately dilute with water to 50 ml. Allow to stand for 2 minute. Compare the colour produced in the two Nessler cylinders.

D-5 RESULT

The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test solution is not greater than that produced in the second Nessler cylinder which is a control test.

ANNEX E

[*Clause* 5.5 and *Table* 1, *Sl No.* (iv)]

DETERMINATION OF ARSENIC

E-1 OUTLINE OF THE METHOD

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.

E-2 REAGENTS

E-2.1 Mixed Acid — Dilute one volume of concentrated sulphuric acid with four volumes of water. Add 10 g of sodium chloride for each 100 ml of the solution.

E-2.2 Ferric Ammonium Sulphate Solution

Dissolve 64 g of ferric ammonium sulphate in water containing 10 ml of mixed acid and make up to one litre.

E-2.3 Concentrated Hydrochloric Acid — *See* IS 265.

E-2.4 Stannous Chloride Solution — Dissolve 80 g of stannous chloride ($SnCl_2.2H_2O$) in 100 ml of water containing 5 ml of concentrated hydrochloric acid.

E-3 PROCEDURE

Carry out the test as prescribed in IS 2088, adding into the Gutzeit bottle, 2 ml of ferric ammonium sulphate solution, 0.5 ml of stannous chloride solution and 25 ml of stannous chloride solution, 0.5 ml of stannous chloride solution and 25 ml of sample solution as prepared in **D-4.1**.

For comparison, prepare a stain using 0.001 mg of arsenic trioxide.

ANNEX F

[*Clause* 5.5 and *Table* 1, *Sl No.* (v)]

DETERMINATION OF FOAMING POWER

F-1 GENERAL

Strict attention shall be paid to all details of the procedure in order to ensure concordant results. Particular care should be taken to shake the cylinder exactly as described.

F-2 OUTLINE OF THE METHOD

A suspension of the material in water is taken in a graduated cylinder and given 12 shakes under prescribed conditions. The volume of the foam formed is observed after keeping the cylinder for 5 minutes.

F-3 APPARATUS

F-3.1 Graduated Cylinder (250 ml), glass stoppered; with graduations from 0 to 250 ml, with 2-ml divisions, overall height about 35 cm and the height of the graduated portion about 20 cm.

F-3.2 Graduated Cylinder (100 ml), with graduations from 0 to 100 ml, with 1 ml divisions.

F-3.3 Thermometer, of range 0 to 110°C.

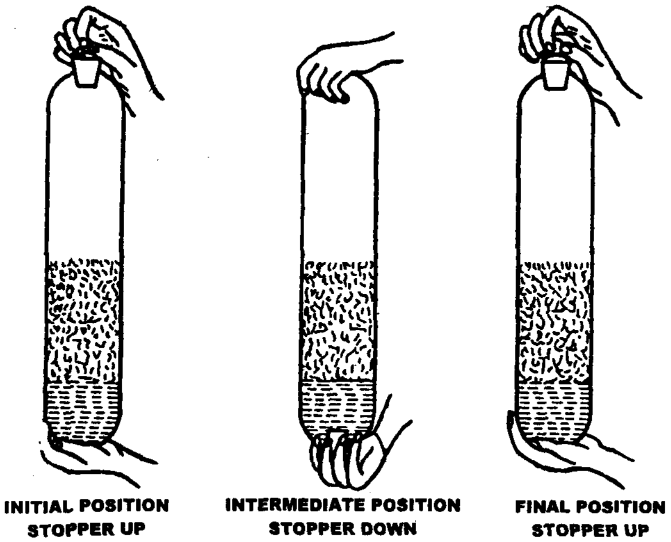
F-4 PROCEDURE

F-4.1 Weigh about 5 g of the toothpaste accurately in a 100-ml glass beaker, add 10 ml of water, cover the breaker with a watch glass and allow to stand for 30 min. This operation is carried out to disperse the toothpaste.

NOTE — Ensure that the detergent is completely dissolved, warming the aqueous suspension, if necessary.

F-4.2 Stir the contents of beaker with a glass rod and transfer the slurry to the 250 ml graduated cylinder, ensuring that no foam (more than 2 ml) is produced and no lump paste goes into the cylinder. Repeat the transfer of the residue left in the breaker with further portions of 5 to 6 ml of water ensuring that all the matter in the beaker is transferred to the cylinder.

F-4.3 Adjust the contents in the cylinder to 50 ml by adding sufficient water and bring the contents of the cylinder to 30°C. Stir the contents of the cylinder with a glass rod or thermometer to ensure a uniform suspension.



INITIAL POSITION
STOPPER UP

INTERMEDIATE POSITION
STOPPER DOWN

FINAL POSITION
STOPPER UP

FIG. 1 ONE COMPLETE SHAKE OF CYLINDER

F-4.4 As soon as the temperature of the contents of the cylinder reaches 30°C, stop the cylinder and give it 12 complete shakes, each shake comprising movements shown in Fig. 1 in a vertical plane, upside down and *vice versa*. After the 12 shakes have been given, allow the cylinder to stand still for 5 minutes and read the volumes of:

a) foam plus water (V_1 ml), and

b) water only (V_2 ml) as shown in Fig. 2.

F-5 CALCULATION

Foaming power, ml = $V_1 - V_2$

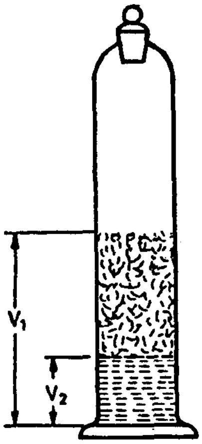


FIG. 2 MEASURMENT OF FOAM

ANNEX G

[*Clause* 5.5, *Table* 1, *Sl No.* (vi)]

DETERMINATION OF FLUORIDE ION

G-1 GENERAL

This method is suitable for the determination of water soluble fluoride species in toothpaste, including free fluoride and hydrolyzable complexes, for example, sodium monofluorophosphate.

G-1.1 Principle

Water soluble species are converted to fluoride ion by acid hydrolysis. The fluoride ion activity is then determined potentiometrically with the help of fluoride ion sensitive electrode.

G-2 Apparatus

G-2.1 *p*H Meter (Potentiometer) — Scale readable to ± 0.5 mV or better.

G-2.2 Fluoride Ion Sensitive Electrode — Orion 94-09 or similar.

G-2.3 Single Junction Reference Electrode — Orion 90-01,or similar, with filling solution.

G-2.4 Magnetic Stirrer

G-2.5 Polythene/Polypropylene Beakers and Volumetric Flasks — 100, 250 ml and pipettes.

G-2.6 Semi-log Graph Papers – 2/3 cycles.

G-3 REAGENTS

G-3.1 Sodium Fluoride (Analytical Grade)

G-3.2 Trisodium citrate (Analytical Grade)

G-3.3 Sodium chloride (Analytical Grade)

G-3.4 Hydrochloric acid (Analytical Grade) — 1M.

G-3.5 Sodium Hydroxide — 1M.

G-3.6 Sodium Acetate Trihydrate (Analytical Grade)

G-3.7 Glacial Acetic Acid

G-3.8 TISAB L (Total Ionic Strength Adjusting Buffer) Solution — Dissolve 294 g trisodium citrate, 29 g sodium chloride and 68 g sodium acetate trihydrate in 600 ml of hot water. Cool, adjust to *p*H 6.4 with glacial acetic acid. Dilute to 1 litre with distilled water.

G-3.9 TISAB LF (TISAB Containing Fluoride) – Solution

Prepare 100 ml of 1mg F^-/100ml solution as described in **G-3.8**. Dissolve 294 g trisodium citrate, 29 g sodium chloride and 68 g sodium acetate trihydrate in 600 ml of hot water. Cool, pipette in 10 ml of 1 mg F^-/100 ml solution and adjust to *p*H 6.4 with glacial acetic acid. Dilute to 1 litre with distilled water. Store in a polythene or polypropylene bottle.

G-3.10 Fluoride Blank Solution

Take 100 ml hydrochloric acid (1M) solution in 1 litre flask and then add 200 ml sodium hydroxide (1M), by measuring cylinder. Dilute to 1 litre with distilled water and mix well.

G-3.11 Standard Sodium Fluoride Solution (0.01 mg F^- per ml)

Dry the sodium fluoride at 110°C for 4 hours and transfer accurately 0.222 g to 100 ml volumetric flask. Add distilled water to dissolve the sodium fluoride and make up to the mark (Solution-X). Each ml of Solution-X contains 1 mg fluoride ion (F^-). Take 10 ml of this Solution-X in 1 000 ml volumetric flask and make up this volume to the mark (Solution-Y). Each ml of Solution-Y contains 0.01 mg fluoride (F^-) ion.

Transfer Solution-X and Solution-Y to polythene bottles for storing.

G-3.12 Preparation of Standard Solutions of Sodium Fluoride

Take 1, 2, 5, 10, 20 and 25 ml of Solution-Y (*see* **G-3.11**) in 100 ml volumetric flask marked A, B, C, D, E and F respectively. To each add 50 ml of TISAB L buffer solution and 10 ml of fluoride blank solution. Check that the *p*H is in the range of 6.4 ± 0.1, and if necessary correct with 1M NaOH or 1 M HCl. Transfer quantitatively to a 100 ml polypropylene volumetric flask and make up the volume to 100 ml with distilled water. Now the solutions A, B, C, D, E and F are containing 0.01, 0.02, 0.05, 0.1, 0.2 and 0.25 mg of F per 100 ml respectively. Transfer the solutions to 150 ml polythene beaker for *mv* measurement.

G-4 mV Measurement of Standard Solutions of Sodium Fluoride

G-4.1 Preparation of Electrodes

Remove protective cap and soak the fluoride electrode in TISAB L F solution for 15 min.

G-4.2 Fill the reference electrode with filling solution.

G-4.3 Rinse the electrodes with de-ionized water and keep the tips immersed in TISAB L F solution until immediately before use.

G-4.4 Check that the electrodes are correctly connected to the *p*H meter.

G-4.5 Rinse the electrodes with deionized water before use and carefully blot dry with a paper tissue.

G-5 mV Measurement

G-5.1 Transfer the contents of solution A from 100 ml flask into a clean, dry 150 ml polypropylene beaker.

G-5.2 Immerse the tips of the electrodes in the solution while stirring the solution with a magnetic stirrer. Ensure that no air bubbles adhere to the electrode surfaces.

G-5.3 Leave until the potential reading is constant. This should take approximately 2 or 3 min.

G-5.4 Record the potential reading in mV and check the temperature of the solution.

G-5.5 Rinse the electrodes with de-ionised water and blot dry with a paper tissue.

G-5.6 Repeat the procedure prescribed in **G-5.1** to **G-5.5** for solutions B, C, D, E and F to record mV of these solutions.

G-5.7 Plot the calibration graph on semi log graph paper with the milli volt reading on the linear ordinate and the final concentration of fluoride in the standard F solution on the logarithmic abscissa. The graph should be a straight line with a gradient of ~ 57-59 mV per decade change in concentration.

G-6 TEST SOLUTION

G-6.1 Discard the first 5 cm of paste extruded from the tube and then weigh about 5 g toothpaste to the nearest mg. Add approximately 30 ml hot (90-95°C) deionized water, a small amount at a time and slurry the paste with a microspatula after each water addition. Allow to cool. Dilute to 100 ml in a polypropylene volumetric flask and mix well.

G-6.2 Ensure that the dispersion is homogeneous and then centrifuge about 60 ml of the dispersion in a polypropylene centrifuge tube, closed with a cap to prevent evaporation, until clear. This will take about 20 min at 4 000 rpm.

G-6.3 Pipette 20 ml of the clear supernatant into a 250 ml round-bottomed flask.

G-6.4 Add a few anti-bumping granules then add 10 ml hydrochloric acid solution (1M approximately) by measuring cylinder. Attach a reflux condenser and boil gently for 5 min.

G-6.5 Add, almost immediately, 20 ml of 1M sodium hydroxide via the condenser, rinsing down with approximate 20 ml of distilled water. Then transfer quantitatively to a 100 ml polypropylene volumetric flask and dilute to volume with distilled water.

G-6.6 Pipette 25 ml of the clear solution prepared above into a 100 ml polypropylene beaker, add 25 ml TISAB L (solution L) and check *p*H. If necessary adjust to *p*H 6.4 by addition of approximately 1M hydrochloric acid or 1M sodium hydroxide. Transfer quantitatively into a 100 ml volumetric flask and dilute to volume.

G-6.7 Transfer the contents of 100 ml flask to a clean, dry 100 ml polythene beaker, immerse the tips of the electrode in the solution while stirring the solution with a magnetic stirrer. Ensure that no air bubbles adhere to the electrode surfaces.

G-6.8 Leave until the potential reading is constant (this should take two or three minutes). Record the potential reading in mV for the test solution.

The reading for standard fluoride and test solutions should be taken simultaneously.

G-7 CALCULATION

A graph is plotted for concentration of F^- against potential mV on a semi-logarithmic paper for standard F solutions. The potential mV is plotted on X-axis and mg of F^- on Y-axis (on logarithmic scale). Read the F concentration in test solution for measured mV from this graph.

$$\text{Concentration of } F^- \text{ in toothpaste, parts per million} = \frac{2a \times 10\,000}{M}$$

where

a = mg of F^- from calibration graph for test solution, and

M = Mass of sample in g.

ANNEX H

(*Foreword* and *Clause* 6)

ABRASIVITY (RDA) MEASUREMENT TEST (HEFFERREN)

H-1 SCOPE

This annex identifies the specific procedures for determination of dentifrice abrasivity using the ADA laboratory method.

H-2 SAMPLING

A representative sample shall be taken from at least two batches.

H-3 PROCEDURE

H-3.1 Standard Reference Abrasive

The standard reference abrasive is from a specific lot of calcium phosphate held by the Monsanto Company.1)

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1) Calcium pyrophosphate is an example of a suitable product available commercially. This information is given for the convenience of users of this standard and does not constitute an endorsement by BIS of this product. A sample of the material may be obtained by contacting the company at the address below and requesting the ADA abrasion standard. Slight shifts (< 10 percent) in abrasivity between lots have been reported.

Monsanto Company
Detergent Division
800 N Lindbergh Boulevard
St Louis MO
USA 63167

H-3.2 Apparatus

H-3.2.1 *Brushing Machine* — A cross brushing machine is the apparatus of choice[1]. The apparatus should have eight positions for holding specimens. A toothbrush shall be positioned to pass reciprocally over the mounted specimens with a designated tension on the brush while immersed in a dentifrice slurry. The distance traversed by the brush should not be longer than the brush head, so that the specimen does not lose contact with the brush. The mechanism for holding the dentifrice slurry may vary with difference machine designs, but should allow for easy removal of the slurry sample. It is important to have some mechanism for agitation of the slurry while the brushing is taking place. A convenient method to accomplish this is to attach rubber mixing vanes just below the brush head. As the brushing takes place, these vanes will prevent the abrasive from settling to the bottom of the slurry container.

H-3.2.2 *Radioactivity Detector* — The two recommended methods for determination of the radioactivity of the used dentifrice slurries are a Gieger-Muller planchet counter or liquid scintillation detector. The use of the Geiger counter requires that the samples be dried under defined controlled conditions. The liquid scintillation method has the advantage of reading directly from the slurry.

Counting should be done for a period expected to reduce the alpha value for counting error to less than 2 percent. Counting should be performed for a minimum of 1 000 counts and at least 1 min. The number of brushing strokes may be increased if counting times become too long.

H-3.3 Presentation of Tooth Specimens

H-3.3.1 *Dentin Specimens*

H-3.3.1.1 *Selection*

Human root dentin of extracted permanent teeth are used as the substrate. Single-rooted teeth that were vital at extraction should be selected. An exception, because of the small size are, mandibular incisors: these should not be used. The specimen should be at least 14 mm long and 2 mm wide at the narrow end. All roots shall be caries-free and free of anatomical defects. After extraction, the roots should be stored in neutralized 4 percent formaldehyde solution.

H-3.3.1.2 *Preparation*

Scrape the roots clean of all soft tissue and cementum. Then remove the crown and the root tips using a separating disc under a flow of water.

H-3.3.1.3 *Irradiation*

For each set of eight specimens to be irradiated, add one or two extra roots for use in correction factors. Pack the specimens in 4 percent formaldehyde solution and submit to a nuclear reactor for irradiation. The neutron flux should be sufficient to produce about 1m Ci of ^{32}P beta radiation after several hours. Elevated temperatures in the reactor should be avoided. It is also advisable to shield the samples from fast neutrons and gamma radiation. Handling of the irradiated specimens should be done with care using good laboratory practice. The specimens should not be used during the first half life because of excess radiation and should be used before the end of the third half-life because of lack of activity. The half-life of ^{32}P is 14.3 days so the usable life-span of a set of teeth is 4 weeks.

H-3.3.1.4 *Mounting of specimens*

The specimens should be mounted individually in cold-cure methyl methacrylate denture resin: the type of mould used will depend upon the holder on the brushing machine. The specimens should be mounted so that they protrude above the resin surface in a buccal/lingual orientation by at least 2 mm. The brushing surface of the root shall be parallel in buccal lingual orientation to the resin holder and situated so that the brushing will take place perpendicular to the long dimension of the root. Storage of the mounted specimens should be in 4 percent formaldehyde.

H-3.3.2 *Enamel Specimens*

H-3.3.2.1 *Selection*

Selection criteria for the enamel specimens are the same as for dentin. The enamel specimens should be obtained from human maxillary incisors.

H-3.3.2.2 *Preparation*

The entire labial surface of the specimen is used after removing the root. Clean the enamel in the same way as the root.

H-3.3.2.3 *Irradiation*

Irradiation of the enamel is identical to the method used with the roots. The roots and enamel specimens may be packed together for submission to the reactor.

H-3.3.2.4 *Mounting*

Mount the enamel specimens in the same way as the roots. The labial surface shall protrude 2 mm and be parallel to the resin surface.

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[1] The drawings and blue prints of the machine may be obtained from the American Dental Association.

H-3.4 *Toothbrushes*

The toothbrushes used should have nylon bristles of medium hardness. The bristle ends should lie in a plane rather than in separated or end-raised tuft design. The bristle length should be about 10 mm. A 50-tuft medium-texture brush shall be used.

Store the brushes in water overnight prior to their first use and then keep them in water until they are discarded. Use a new set of brushes for each set of teeth. Do not remove the brushes from the machine between runs but raise the tufts of the specimen so as not to bend the bristles. At the beginning of each run, set the tension, of the brush on the specimen to 150 g using a Chatilon spring gauge or equivalent. This tension should be rechecked at least twice daily. The method of adjusting the tension will vary depending upon the type of mechanism on the brushing machine.

H-3.5 Reference Diluent

The diluent is a 0.5 percent carboxymethylcellulose (CMC) (7MF) solution in 10 percent glycerine. To prepare 1 litre of the diluent, heat 50 ml of glycerine to 60°C and add 5g of CMC while stirring. After the mixture is homogeneous, add another 50 ml of heated glycerine and continue stirring for 60 min. Transfer the solution to a 1 litre flask and add 900 ml of distilled water. Allow to cool but continue stirring slowly overnight. To stabilize the viscosity, allow the solution to stand overnight before using. This solution is used to make up slurries of the reference abrasive or any other powder being tested.

H-3.6 Reference Abrasive Slurry

The reference material is that described above (*see* **H-3.1**). Dilute 10 g of the abrasive with 50 ml of the diluent (*see* **H-3.5**). The same ratio is used for all powders. It is possible for the reference abrasive to be used as a dentifrice, if that has to be done. It shall be made up as a 40 percent abrasive dentifrice with the rest of the constituents being conventional dentifrice components. The slurry is then made with 25 g of reference dentifrice and 40 ml of water.

H-3.7 Dentifrice Slurries

To prepare the test slurries, add 40 ml of water to 25 g of each dentifrice. For the machine, prepare eight slurries of each dentifrice. This dilution produces a final slurry volume and concentration similar to those of the reference abrasive slurry. All slurries reference and test should be used shortly after preparation and after vigorous mechanical stirring to prevent particle setting.

H-3.8 Preconditioning of Tooth Specimens

H-3.8.1 *Dentin*

To reduce the variation caused by dentin surface differences, precondition the specimens prior to each use. The preconditioning treatment consists of brushing with slurry of the reference abrasive but not taking a sample. The first time dentin specimens are used, the preconditioning should be for 6 000 strokes. Each successive daily run should begin with a shorter precondition brushing of 1 000 strokes. The tension of the toothbrush on the roots shall be 150 g.

Discard the preconditioning slurries.

H-3.8.2 *Enamel*

Preconditioning of the enamel is similar to that of the dentin, except 10 000 strokes are used prior to the first use and 1 000 strokes are given at the beginning of each day.

Discard the preconditioning slurries.

H-3.9 Test Design

H-3.9.1 *Test Design for Dentin*

The test design may be either a sandwich design or a Latin square design. The sandwich design is such that a set of reference slurries is run (pre-test), followed by a second set of reference slurries (post-test).This second set of reference slurries (post-test). This second set of reference slurries then acts as the pre-test slurries for the next test group. This continues until all the test groups are run.

The Latin square design is such that a set of reference slurries is run first. All the test groups are randomized over the eight brushing heads for the next few runs (depending on the number of test groups). Then a post-test reference set of slurries is run as the final procedure.

In both test designs, the brush tension is set at 150 g and brushing is performed for 1 500 to 3 000 strokes depending on the radioactivity level of the specimens.

H-3.9.2 *Test Design for Enamel*

The test design for enamel is identical to that for dentin, except the number of strokes is 5 000 to 7 500 depending on the activity of the specimens.

H-3.10 Sampling of Slurries

The sampling of the slurries following the brushing is identical for both dentin and enamel. An eliquot of each slurry is removed immediately following brushing. The size of the aliquot will depend upon the counting method and equipment, but 3 ml is usually adequate to provide a detectable level of radioactivity. A convenient method for removing the sample is a syringe fitted with a blunt needle. Take care to ensure no carry-over between samples. This can best be done by a complete rinsing of the syringe between samples. It is also important to remove the same quantity of sample from each slurry. Dry the sample if a planchet counter system if being used to detect the radioactivity. If drying is needed, the samples should be air-dried for at least 1 h and then dried in an oven at 60°C with forced air overnight.

H-3.11 Correction Factors

Correction factors are needed for both dentin and enamel abrasion tests when using the planchet counting method and are identically prepared in both methods. When testing dentifrices with abrasive systems different from the reference materials, the self-absorption and backscattering characteristics of the abrasives for beta radiation may also differ. Real differences in abrasivity may then be significantly distorted. The correction factor is a means to reduce this variable. The correction factor is determined differently depending on the counting method used.

H-3.11.1 *Preparation of Correction Factor Slurries for Geiger-Muller Planchet Counting*

Dissolve one piece of irradiated dentin (or enamel) in 5 ml of concentrated HCl. Transfer the solution to a 250 ml volumetric flask and add water to the mark. Add 1.0 ml of this radioactive solution to slurries of the reference abrasive and to each of the test abrasives prepared in the same manner as in the test. To neutralize the acid, add 1.0 ml of 0.5 mol/NaOH. Mix the slurries thoroughly, sample and dry the samples along with those from the test runs. Do not brush with these correction factor slurries.

These samples are counted along with the test samples.

H-3.11.2 *Calculation of Correction Factors*

The correction factor *Cf* to be applied to all values of the test sample is calculated as follows:

$$Cf = \frac{\text{Mean counts for 4 reference samples}}{\text{Mean counts for 4 test samples}}$$

H-3.11.2.1 *Correction factors for liquid scintillation counting*

The correction is with regard to the amount of sample mixed with the scintillation cocktail. Each sample is weighed and the net count per minute (CPM) is divided by the mass to get a net CPM per gram of slurry. These net CPM per gram of values are then used in calculating abrasivity in place of net CPM values in **H-3.12**, and there is no *Cf* term.

H-3.11.2.2 *Correction factors for liquid scintillation detection*

Self-absorption and backscatter are less of a concern because of the liquid medium being used. Most modern liquid scintillation equipment will automatically colour-correct, so this is not a problem. The differences in mass of the samples do need to be accounted for in the calculation. To do this, each sample taken after brushing needs to be weighed to an accuracy of 0.01g.

H-3.11.2.3 *Applying the correction factor*

Before calculating the relative abrasion values, the net CPM of each slurry is divided by the mass of the slurry used, to get a net CPM per gram of slurry. These values are then used in the calculation of relative abrasive values.

H-3.12 Calculation of Abrasivity Using Gieger-Muller Counting

H-3.12.1 *Dentin Abrasivity*

The dentin abrasivity of the test dentifrices (or abrasives) is calculated as follows:

$$\text{Mean Reference net CPM} = \frac{\text{Pre-net CPM} + \text{Post-net CPM}}{2}$$

$$\text{Dentifrice abrasivity} = \frac{Cf \times 100 \times \text{test dentifrice Net CPM}}{\text{Mean reference net CPM}}$$

H-3.12.2 *Enamel Abrasivity*

The enamel abrasivity of the test dentifrices for abrasives is calculated as follows:

$$\text{Mean reference net CPM} = \frac{\text{Pre-net CPM} + \text{Post-net CPM}}{2}$$

$$\text{Dentifrice abrasivity} = \frac{Cf \times 10 \times \text{test dentifrice Net CPM}}{\text{Mean reference net CPM}}$$

H-3.13 Calculation of Abrasivity using Liquid Scintillation

H-3.13.1 *Dentin Abrasivity*

The dentin abrasivity of the test dentifrices (or abrasives) is calculated as follows:

$$\text{Mean reference net CPM per gram} = \frac{\text{Pre-net CPM per gram} + \text{Post-net CPM per gram}}{2}$$

$$\text{Dentifrice Abrasivity} = \frac{100 \times \text{Test dentifrice net CPM per gram}}{\text{Mean reference net CPM per gram}}$$

H-3.13.2 *Enamel Abrasivity*

The enamel abrasivity of the test dentifrices (or abrasives) is calculated as follows:

$$\text{Mean reference net CPM per gram} = \frac{\text{Pre-net CPM per gram} + \text{Post-net CPM per gram}}{2}$$

$$\text{Dentin abrasivity} = \frac{10 \times \text{test dentifrice net CPM per gram}}{\text{Mean reference net CPM per gram}}$$

NOTE

Centres for measuring toothpaste abrasivity via RDA route:

1. Indiana University , School of Dentistry
   Oral Health Research Institute
   415 Lansing Street
   Indianapolis, Indiana 46202
   USA
2. Missouri Analytical grade Laboratories
   Marcus Research Laboratories, INC
   1820 Delmar Boulevard
   St Louis, MO 63103-1798
   USA

ANNEX J

(*Clause* 2)

LIST OF ADJUNCT INDIAN STANDARDS

IS No.	*Title*
918 : 1985	Calcium carbonate precipitated for cosmetic industry (*second revision*)
1070 : 1992	Water reagent grade (*third revision*)
1463 : 1983	Kaolin for cosmetic industry (*third revision*)
1767 : 1980	Dicalcium phosphate for dentifrice (*second revision*)
1796 : 1986	Glycerol (*second revision*)
2124 : 1974	Sodium bicarbonate (*first revision*)
2528 : 1984	Magnesium carbonate for cosmetic industry (*third revision*)
3401 : 1992	Silica gel (*third revision*)
3986 : 1988	Sodium lauryl sulphate for cosmetic industry (*third revision*)
3987 : 1983	Sorbitol solution (70%) (*second revision*)
3988 : 1989	Guar gum (*first revision*)
5025 : 1988	Gum karaya (*second revision*)
5191 : 1993	Sodium alginate, food grade (*first revision*)
5306 : 1996	Sodium carboxy methyl cellulose, food grade (*second revision*)
5345 : 1996	Sodium saccharin, food grade (*second revision*)
5719 : 1994	Gelatine, food grade
7238 : 1997	Gum tragacanth, food grade (*first revision*)
10563 : 1990	Mineral oil, food grade
13657 : 1993	Aspartame, food grade
13702 : 1993	Propylene glycol, food grade

ANNEX K

(*Foreword*)

COMMITTEE COMPOSITION

Cosmetics Sectional Committee, PCD 19

Organization	*Representative(s)*
Directorate General of Health Services, New Delhi	Shri Ashwini Kumar (***Chairman***)
All India Small Scale Cosmetic Manufacturer's Association, Mumbai	Shri M. B.Desai Shri B. M. Chopra (*Alternate* I) Shri S. Chatterjee (*Alternate* II)
Bengal Chemicals & Pharmaceuticals Ltd, Kolkata	Dr Sajal K. Roy Chowdhury Dr A. K. Mandal (*Alternate*)
Central Drugs Laboratory, Kolkata	Dr M. K. Mazumder Dr A. C. Das Gupta (*Alternate*)
Central India Pharmacopoeia Laboratory, Ghaziabad	Dr Santosh. K. Talwar Dr Sukomal Das (*Alternate*)
Consumer Education and Research Centre, Ahmedabad	Dr C. J. Shishoo Shri Y. S. Yellore (*Alternate*)
Consumer Guidance Society, Mumbai	Shri. N. G. Wagle Dr S. G. Bhat (*Alternate*)
Colgate-Palmolive (India) Ltd, Mumbai	Dr Raj Kohli Shri Sunil Aggarwal (*Alternate* I) Dr Neena Sharma (*Alternate* II)
Commissioner, Food & Drugs Administration, Mumbai	Dr Pilankar
Dabur Research Foundation, Sahibabad	Dr D. B. A. Narayana
Food & Drugs Control Admn Gujarat State, Gandhinagar	Dr P. J. Thomas Shri J. J. Shukla (*Alternate*)
Godrej Soaps Ltd, Mumbai	Shri A. Rangarajan Dr (Shrimati) Rohini Thakkar (*Alternate*)
Hindustan Lever Research Centre, Mumbai	Dr Pushker Sona, Shri N. S Bijlani (*Alternate* I) Shri Cyrus Dalal (*Alternate* II)
Hygienic Research Institute, Mumbai	Shri M. B. Desai Shri Manish K. Chhabra (*Alternate*)
Indian Soaps and Toiletries Members Association, Mumbai	Shri V. P. Menon
In personal capacity	Dr S. N. Iyer
Johnson & Johnson Ltd, Mumbai	Dr Prashant Abhyankar Dr V. R. Bambulkar (*Alternate*)
Lady Irwin College, New Delhi	The Principal
Lady Amritbai Doga College, Nagpur	Dr (Shrimati) S. B. Kulkarni
Maharishi Ayurved Products, Noida, U. P.	Dr S. C. Saxena Shri V. K. Shrivastava (*Alternate*)
Nahira Cosmetic Enterprises Pvt Ltd, Mumbai	Shri Madhukar Y. Bordawekar Shri G. J. Lad (*Alternate*)
National Test House, Kolkata	Shri K. C. Naskar Dr S. K. Saha (*Alternate*)
Procter & Gamble, Mumbai	Dr Arun Viswanath Ms Shweta Purandare (*Alternate*)
Shingar Ltd, Mumbai	Shri V. K. Singh Shrimati Varsha Bisen (*Alternate*)

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Organization	*Representative(s)*
Shriram Institute for Industrial Research, New Delhi	Shri S. K. Chib Dr U. C. Bahri (*Alternate*)
BIS Directorate General	Shri Anjan Kar, Director & Head (PCD) [Representing Director General (*Ex-officio*)]

Member-Secretary

Dr (Shrimati) Vijay Malik

Director (PCD), BIS

Miscellaneous Cosmetics Products Subcommittee, PCD 19:5

Colgate Palmolive (India) Ltd Mumbai	Dr Raj Kohli (*Convener*) Dr Neena Sharma (*Alternate* I) Shri Sunil Agrawal (*Alternate* II)
Central Indian Pharmacopoeia Laboratory, Ghaziabad	Dr Sukomal Das Shri H. S. Goel (*Alternate*)
Consumer Guidance Society, Mumbai	Shri N. G. Wagle
Dabur Research Foundation, Sahibabad	Dr D. B. A. Narayana Shri Dhiraj Khattar (*Alternate*)
Food & Drug Administration, Mumbai	Dr Pilankar
Geoffrey Manners & Co Ltd, Mumbai	Shri B. D. Kale Shri C. S. R. Kulshreshtra (*Alternate*)
Godrej Soaps Ltd, Mumbai	Shri A. Rangarajan Dr (Shrimati) Rohini Thakkar (*Alternate*)
Hindustan Lever Research Centre, Mumbai	Dr Pushker Sona Shri S. M. Shanbhag (*Alternate*)
Nogi & Co Pvt Ltd, Mumbai	Dr R. D. Pandit Shri M. S. Ambardekar (*Alternate*)
Procter & Gamble, Mumbai	Ms Shweta Purandare Dr Arun Viswanath (*Alternate*)
Shriram Institute for Industrial Research, New Delhi	Shri K. M. Chacko Shri A. K. Gupta (*Alternate*)

(*Continued from second cover*)

certain conditions excessive ingestion of fluoride may contribute to fluorosis. Keeping both the aspects in mind, the Ministry of Health and Family Welfare has imposed a restriction on limit of fluoride ion in toothpaste. Therefore, fluoride ion in a fluoridated toothpaste should not exceed the limit prescribed in this specification.

While retaining the maximum available fluoride level at 1 000 ppm for fluoridated toothpaste, a simpler method for fluoride analysis has been included in this standard.

While no changes have been made on the maximum limit on heavy metal content expressed as lead in toothpaste, a safer method has been adopted for the estimation of heavy metals.

The test protocol for measuring microbial counts in toothpaste has been modified in line with the methods stipulated in IS 14648 : 1999 'Methods of test for microbiological examinations of cosmetics'. Tests for gram negative pathogens as a group are included and specific testing for *Salmonellae* and *Escherichia coli* has been discontinued since these organisms are part of this group.

No stipulations have been made regarding the composition of toothpastes, however, it is essential that the toothpaste formulations do not contain any ingredient in sufficient concentration to cause a toxic or irritating reaction when used in the mouth. Nor shall it be harmful in normal use, keeping in mind that small amounts may be ingested inadvertently. Certain new ingredients for guidance have been included in the light of new developments in oral care formulations.

Stipulation has been made for declaration by the manufacturer in the form of 'Best use before' (month and year) to be mentioned on tube and carton for all the types of toothpaste along with the list of key ingredients.

A scheme for labelling environment friendly products known as ECO-Mark (optional) has been introduced at the instance of the Ministry of Environment and Forests (MEF), Government of India. The ECO-Mark is being administered by the *Bureau of Indian Standards Act*, 1986 as per the Resolution No. 71 dated 21 February 1991 and No. 768 dated 24 August 1992 published in the Gazette of the Government of India. For a product to be eligible for marking with ECO logo, it shall also carry the Standard Mark of BIS besides meeting additional environment friendly requirements. For this purpose, the Standard Mark of BIS would be a single mark being a combination of the BIS monogram ISI and the ECO logo. Requirements for the ECO friendliness will be additional, manufacturing units will be free to opt for Standard Mark alone also.

Composition of the Committee responsible for the formulation of this standard is given in Annex K.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc: No. PCD 19 (1747).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 323 01 31, 323 3375, 323 94 02

Telegrams: Manaksanstha
(Common to all offices)

Regional Offices:

Regional Offices	Address	Telephone
Central	Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	323 76 17, 323 38 41
Eastern	1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi CALCUTTA 700054	337 84 99, 337 85 61 337 86 26, 337 91 20
Northern	SCO 335-336, Sector 34-A, CHANDIGARH 160022	60 38 43 60 20 25
Southern	C.I.T. Campus, IV Cross Road, CHENNAI 600113	254 12 16, 254 14 42 254 25 19, 254 13 15
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