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Dentistry — Toothpastes — Requirements, test methods and marking

Produits et matériel pour l'art dentaire — Dentifrice — Prescriptions, méthodes d'essai et marquage

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11609 was prepared by Technical Committee ISO/TC 106, *Dentistry*, in collaboration with the International Dental Federation (FDI).

Annex A forms an integral part of this International Standard. Annexes B and C are for information only.

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Dentistry — Toothpastes — Requirements, test methods and marking

1 Scope

This International Standard specifies requirements and test methods for physical and chemical properties, and for the marking and/or labelling of toothpastes for daily use by the public with a toothbrush to promote oral hygiene.

NOTE 1 It is anticipated that guidelines on claimed or implied efficacy of toothpastes for the prevention or control of oral conditions will be published as an FDI Technical Report.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1942:1989, *Dental vocabulary.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

BS 5136:1981, *Specification for toothpastes.*

CTFA, *International Cosmetic Ingredients Dictionary.*[1)]

SHELLIS, R.P. A synthetic saliva for cultural studies of dental plaque. *Archives Oral Biol.*, **23**, pp. 485-489, 1978.

POPE, D.G. Accelerated stability testing for production of drug product stability. *Drugs and Cosmetics,* pp. 54-62, 1980.

3 Definitions

For the purposes of this International Standard, the definitions given in ISO 1942 and the following definitions apply.

3.1 dentifrice: Any substance or combination of substances specially prepared for the public for cleaning the accessible surfaces of teeth.

3.1 toothpaste: Any semi-solid dentifrice preparation presented in the form of a paste, cream or gel.

NOTE 2 The product's main constituents generally are an abrasive, humectants, a binder, a surfactant and a flavouring. Secondarily, the product may contain agents for oral health maintenance.

4 Requirements

4.1 Total fluoride

The total fluoride concentration shall not exceed the limits set by national laws and regulations and in no case shall the total fluoride concentration exceed 0,15 % (*m/m*). When using containers, for example dispensing systems of various sorts, the total fluoride content per single unit shall not exceed 300 mg.

Test in accordance with one of the procedures given in annex B or the EEC method[22]. Other validated methods of similar sensitivity and accuracy may be used (see references [20] and [21].

1) See note 4, clause 6.

4.2 Heavy metals

The maximum concentration shall not exceed the limits set by national laws and regulations. In no case shall the total heavy metal concentration in the toothpaste be greater than 20 mg/kg.

Test in accordance with reference [10], [11] or [12], or other validated method of similar sensitivity and accuracy.

4.3 Alkalinity

When tested in accordance with 5.2.1, the toothpaste shall have a pH below 10,5.

4.4 Demineralization

Toothpastes having a pH of less than 5,5 shall be tested for demineralization in accordance with 5.2.2. The enamel lost from teeth exposed to the test shall not exceed that lost from teeth exposed to the control treatment or shall comply with the requirements of the enamel abrasion test (5.2.2.2).

4.5 Compatibility with oral tissues

The toothpaste should not cause irritation or damage to the oral tissue when used as intended by the manufacturer.

Test in accordance with 5.2 and 5.3.

NOTE 3 Specific qualitative and quantitative requirements for freedom from biological hazards are not included in this International Standard, but it is recommended that reference should be made to ISO/TR 7405[1] and ISO 10993-1[2] when assessing possible biological or toxicological hazards.

4.6 Microbiology

Testing for microbiological contamination shall be carried out according to reference [9] or references [3], [4] and [5].

4.7 Abrasivity

The abrasivity of the toothpaste shall not exceed the following limits for dentin:

— 2,5 times, if using the procedure specified in annex A, or

— 2 times, if using the procedure specified in BS 5136,

that of the primary reference material.

Test in accordance with 5.3 or any other validated method of similar sensitivity and accuracy.

4.8 Stability

The toothpaste shall show no signs of deterioration which may affect compliance with this International Standard after being subjected to the ageing procedure specified in 5.4.

4.9 Readily fermentable carbohydrates

The toothpaste shall not contain readily fermentable carbohydrates. Compliance shall be established by the absence of such compounds in the complete formula or by performing tests in accordance with commonly used analytical methods.

5 Test methods

5.1 Visual inspection

The toothpaste shall show no signs of deterioration (e.g. separation, discoloration, viscosity/extension difficulties) after being stored according to the accelerated ageing procedures specified in 5.4.1. This attests to its compliance with this International Standard. Examine under a bright light with normal visual acuity without magnification.

5.2 Determination of pH and enamel demineralization

5.2.1 Determination of pH

Suspend one part by mass of the toothpaste into three parts by mass of water for analytical laboratory use complying with ISO 3696 (grade 2). Determine the pH of the suspension within 10 min, using a pH-meter with a glass and calomel electrode assembly.

5.2.2 Determination of enamel demineralization or abrasion (low pH compositions)

The toothpaste shall comply with the requirements of either 5.2.2.1 or 5.2.2.2.

5.2.2.1 Enamel demineralization

For toothpaste with a pH below 5,5, use the following procedure.

Prepare radioactive human enamel (see annex A or B) in a manner that will provide a uniform surface area on all specimens. Perform the irradiation of the specimens in accordance with the procedure for radioactive abrasion tests. Cover the remainder of the enamel with an acid-resistant varnish.

Immerse the specimens in artificial saliva for 1 h to promote a slight protein film simulating oral condi-

tions. Immerse the specimens individually in 20 ml of 1:3 slurries of the test toothpaste in artificial saliva. Stir the slurries constantly and uniformly; maintain continual immersion.

Use double-distilled or deionized water (ISO 3696) as a negative control.

After immersion for 24 h analyse the slurries for the level of radioactivity (^{32}P). To pass the test, the enamel lost to the slurries shall not be more than that lost to the water control.

5.2.2.2 Enamel abrasion

If the pH of the toothpaste is below pH 5,5, then it shall not remove more than four times the level of human enamel compared to that removed by BS 5136 toothpaste modified as follows: use the toothpaste as a 3:1 artificial saliva/toothpaste slurry. If tested using the Hefferren procedure (annex A), the toothpaste shall not remove more than twice the amount of enamel removed by the reference abrasive when both the toothpaste and the reference abrasive are used as a 3:1 artificial saliva/toothpaste/abrasive slurry. Test in accordance with annex A or BS 5136. Other validated methods of similar sensitivity and accuracy may be used.

5.3 Determination of abrasivity

Determine the mean relative abrasivity compared to the primary reference sample, or any other reference material calibrated to the primary reference sample for human dentin, using the method specified in annex A or BS 5136.

Other validated methods of similar sensitivity and accuracy may be used; see for example references [14], [15] and [16].

5.4 Determination of stability

5.4.1 Accelerated ageing procedure

The toothpaste shall meet the requirements of this International Standard after storage at 40 °C for 3 months or under such conditions of time and temperature as will simulate storage at room temperature for 30 months.

5.4.2 Shelf-life

The toothpaste shall meet the requirements of this International Standard 30 months after manufacture.

5.4.3 Container and/or dispensing system

The container and/or dispensing system shall not have contaminated the toothpaste inside so as to affect its compliance with this International Standard after being subjected to the ageing procedure described in 5.4.1.

6 Marking and labelling

With the exception of dispensing systems and small single units, packaging shall be marked with the following information:

a) the word "toothpaste" or equivalent as defined in 3.2;

b) the brand name;

c) name and address of manufacturer or responsible distributor;

d) batch or lot number;

e) a list of ingredients: a complete declaration according to the *CTFA International Cosmetic Ingredients Dictionary*, or with descriptive names of ingredients; identification of ingredients shall be consistent with the dictionary, which states how the declaration should be made and the ingredients identified;

f) net volume, in millilitres, and/or net mass, in grams;

g) expiration date, if the period of stability (shelf-life) is less than 30 months.

NOTE 4 After 1997, CTFA guidelines will be succeeded by INCI guidelines (International Nomenclature for Cosmetic Ingredients).

Annex A
(normative)

Abrasivity test procedure (Hefferren)

A.1 Scope

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

A.2 Sampling

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

A.3 Procedure

A.3.1 Standard reference abrasive

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

A.3.2 Apparatus

A.3.2.1 Brushing machine

A crossbrushing machine is the apparatus of choice.[2)] 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 different 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.

A.3.2.2 Radioactivity detector

The two recommended methods for determination of the radioactivity of the used dentifrice slurries are a Geiger-Müller 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 %. 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.

A.3.3 Presentation of tooth specimens

A.3.3.1 Dentin specimens

A.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 % formaldehyde solution.

A.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.

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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 International Standard and does not constitute an endorsement by ISO 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 %) in abrasivity between lots have been reported.

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

2) The drawings and blueprints for the machine may be obtained from the American Dental Association.

A.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 % formaldehyde solution and submit to a nuclear reactor for irradiation. The neutron flux should be sufficient to produce about 1 mCi 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.

A.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 % formaldehyde.

A.3.3.2 Enamel specimens

A.3.3.2.1 Selection

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

A.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.

A.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.

A.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.

A.3.4 Toothbrushes

The toothbrushes used should have nylon bristles of medium hardness. The bristle ends should lie in a plane rather than in serrated or end-raised tuft design. The bristle length should be about 10 mm. A 50-tuft medium-texture Pepsodent brush is a commercially available brush that meets acceptable criteria.[3)]

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 off 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 Chatillon 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.

A.3.5 Reference diluent

The diluent is a 0,5 % carboxymethylcellulose (CMC) (7MF) solution in 10 % glycerine. To prepare 1 l of the diluent, heat 50 ml of glycerine to 60 °C and add 5 g 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 l 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 powder being tested.

A.3.6 Reference abrasive slurry

The reference material is that described above (A.3.1). Dilute 10 g of the abrasive with 50 ml of the diluent (A.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 is to be done, it shall be made up as a 40 % 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.

A.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

3) This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named.

and test) should be used shortly after preparation and after vigorous mechanical stirring to prevent particle settling.

A.3.8 Preconditioning of tooth specimens

A.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 a 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.

A.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.

A.3.9 Test design

A.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 set of the first test slurries. They are then followed by a 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.

A.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.

A.3.10 Sampling of slurries

The sampling of the slurries following the brushing is identical for both dentin and enamel. An aliquot 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 is 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.

A.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 material, 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.

A.3.11.1 Preparation of correction factor slurries for Geiger-Müller 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/l 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.

A.3.11.2 Calculation of correction factors

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

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

A.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 A.3.12, and there is no Cf term.

A.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 0,01 g.

A.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.

A.3.12 Calculation of abrasivity using Geiger-Müller counting.

A.3.12.1 Dentin abrasivity

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

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

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

A.3.12.2 Enamel abrasivity

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

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

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

A.3.13 Calculation of abrasivity using liquid scintillation

A.3.13.1 Dentin abrasivity

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

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

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

A.3.13.2 Enamel abrasivity

The enamel abrasivity of the test dentifrice (or abrasive) is calculated as follows:

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

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