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Dentistry — Dentifrices — Requirements, test methods and marking

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 7, *Oral care products*.

This third edition cancels and replaces the second edition (ISO 11609:2010), which has been technically revised.

Introduction

Dentifrices should not cause any adverse reactions to the oral soft tissues when used in accordance with the manufacturer's recommendation for frequency and duration of use, nor cause any known side effects.

Guidelines on assessing the claimed or implied efficacy of dentifrices for the prevention or control of oral conditions can be found through the US Food and Drug Administration[3], the American Dental Association[4] and the Commission Work Project (8-95) of the FDI World Dental Federation[16].

Dentistry — Dentifrices — Requirements, test methods and marking

1 Scope

This document specifies requirements for the physical and chemical properties of dentifrices and provides guidelines for suitable test methods. It also specifies requirements for the marking, labelling and packaging of dentifrices.

This document applies to dentifrices, including toothpastes, destined to be used by the consumers on a daily basis with a toothbrush to promote oral hygiene.

Specific qualitative and quantitative requirements for freedom from biological and toxicological hazards are not included in this document. These are covered in ISO 7405[1] and ISO 10993-1[2].

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.

ISO 1942, *Dentistry — Vocabulary*

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

ISO 8601, *Data elements and interchange formats — Information interchange — Representation of dates and times*

International Nomenclature of Cosmetic Ingredients (INCI), *in International Cosmetic Ingredient Dictionary and Handbook*[1)]

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1942 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
dentifrice
any substance or combination of substances specially prepared for the consumers for hygiene of the accessible surfaces of teeth and surrounding tissues

3.2
toothpaste
any semi-solid dentifrice preparation presented in the form of a paste, cream or gel

Note 1 to entry: The product's common constituents are abrasives, humectants, binders, surfactants, flavourings, fluorides and other agents for oral health benefits.

1) Nomenclature developed by the Personal Care Products Council (formerly CTFA). Available at: https://access.personalcarecouncil.org/eweb/DynamicPage.aspx?Site=pcpc&WebKey=4513b14e-2f75-4857-85b4-b3697be5d5d9.

3.3
single-unit container
container of dentifrice marketed to individual consumers

3.4
primary container
container that is in direct contact with the product

4 Requirements relative to the physical and chemical properties of dentifrices

4.1 Total fluoride

4.1.1 Total fluoride concentration

The total fluoride concentration shall not exceed a mass fraction of 0,15 % when tested in accordance with one of the procedures given in Annex C.

Other validated methods of similar sensitivity and accuracy may be used (see References [5] to [12], [28] and [29]).

4.1.2 Total fluoride in a single-unit container

The amount of total fluoride in a single-unit container shall not exceed 300 mg.

This requirement does not apply to containers of dentifrice to be dispensed under professionally supervised conditions or in community-based caries prevention programmes such as school toothbrushing programmes.

4.2 Heavy metals

The total maximum concentration of heavy metals shall not exceed 20 mg/kg.

Test in accordance with References [13], [14] or [15], or another validated method of similar sensitivity and accuracy.

4.3 pH

When tested in accordance with 5.1, the dentifrice shall have a pH below 10,5.

4.4 Microbiology

Testing for microbiological contamination shall be carried out according to References [17] to [22] and [31] to [38] or any other validated method of equivalent sensitivity, accuracy and specificity.

4.5 Abrasivity

The abrasivity of the dentifrice shall not exceed the following limit for dentine:

— 2,5 times that of the primary reference material, if using the procedure specified in Annex A or B;

The abrasivity of the dentifrice shall not exceed the following limit for enamel:

— four times that of the primary reference material, if using the procedure specified in Annex A or B.

Test in accordance with 5.2 or 5.3 or any other validated method of similar sensitivity and accuracy.

4.6 Stability

The dentifrice shall show no deterioration that may affect compliance with this document or could result in toxicological hazards after being subjected to one of the ageing procedures specified in 5.4 or after 30 months of storage at room temperature. If deterioration is detected, the dentifrice shall be labelled with an expiry date.

4.7 Readily fermentable carbohydrates

The dentifrice 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 Determination of pH

Suspend one part by mass of the dentifrice into three parts by mass of water for analytical laboratory use complying with ISO 3696 (grade 3). Determine the pH of the suspension within 10 min, using a pH-meter and electrode assembly.

5.2 Determination of dentine 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 dentine, using one of the methods specified in Annex A or B.

Other validated measurement methods on dentine of similar sensitivity and accuracy may be used, conforming to practices and principles found in References [39] to [44]. For other references see, for example, References [23] and [24].

5.3 Determination of enamel 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 enamel, using one of the methods specified in Annex A or B.

Other validated measurement methods on enamel of similar sensitivity and accuracy may be used, conforming to practices and principles found in References [39] to [44]. For other references see, for example, References [23] and [24].

5.4 Determination of stability

For the accelerated ageing procedure, the dentifrice shall be stored in its original container at 40 °C ± 2 °C at 75 % ± 5 % relative humidity for 3 months or at such conditions of time and temperature as will simulate storage at room temperature for 30 months[25]. Following storage, test the product according to this document.

6 Marking and labelling

With the exception of small single units (less than 10 ml), all primary containers shall be marked with the following information:

a) the word “dentifrice” or equivalent (see Clause 3);

b) the trade name;

c) the name and contact information of the manufacturer or responsible distributor;

d) the tracking code that includes an intelligible production date;

e) a complete list of ingredients according to the International Nomenclature of Cosmetic Ingredients (INCI);

f) the concentration and type of fluoride, if present, expressed in micrograms per gram, or percent by mass, or both;

g) the net volume, in millilitres, or net mass in grams, or both;

h) the expiry date, expressed according to ISO 8601, if the period of stability (shelf-life) is less than 30 months;

i) a safety notice regarding the use, by children below 6 years of age, of dentifrices containing concentrations of fluoride of 1 000 µg/g or more.

7 Packaging

The product shall be packaged in such a way that under normal conditions of handling and transport, the container or dispensing system, or both, shall not contaminate or permit contamination of the dentifrice inside, so as to affect its compliance with this document, after being subjected to the ageing procedure described in 5.4.

Annex A
(informative)

Abrasivity test procedure — American Dental Association (ADA) method

A.1 General

This annex identifies the specific procedures for determination of the dentifrice abrasivity using the ADA laboratory method[26].

A.2 Sampling

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

A.3 Procedure

A.3.1 Standard reference abrasive

The primary reference abrasive is from a specific lot of calcium pyrophosphate[2]. An alternate, silica reference abrasive[3], is also available[27]. For the procedure specified in BS 5136[30], a chalk reference dentifrice is also available.

A.3.2 Apparatus

A.3.2.1 Brushing machine.

A cross-brushing machine is the apparatus of choice[4]. The apparatus should have eight positions for holding specimens. A toothbrush shall be positioned to pass reciprocally at a small angle (approximately 5°) 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 the 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.

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2) Reference calcium pyrophosphate is available from Odontex Inc. 3030 Campfire Dr., Lawrence, KS 66040, USA, http://www.odontexusa.com. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

3) Alternative reference silica (Sident®) is available from Evonik, Rodenbacher Chaussee 4, 63457 Hanau Wolfgang, Germany. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

4) An acceptable product is available from Sabri Dental Enterprises, Inc., 1404 Brooke Dr., Downers Grove, IL 60515, USA, http://www.sabridentalenterprises.com/p/about.html. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

A.3.2.2 Radioactivity detector.

The two recommended methods for the determination of the radioactivity of the used dentifrice slurries are a Geiger-Müller planchet counter and a 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 for at least 1 min. The number of brushing strokes may be increased if counting times become too long.

A.3.3 Preparation of tooth specimens

A.3.3.1 Dentine specimens

A.3.3.1.1 Selection

Human root dentine of extracted permanent teeth is 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 a neutralized solution that disinfects but does not alter the physical properties.

A.3.3.1.2 Preparation

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

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 disinfection 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 (above 65 °C) should be avoided. A specific position shall be requested 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

Mount the specimens individually in a mould in cold-cure methyl methacrylate resin such that either the buccal or lingual surface protrudes at least 2 mm above and parallel to the resin. Orient the mould in the brushing machine such that the direction of brushing is perpendicular to the long dimension of the root. Store the mounted specimens in a neutralized solution that disinfects but does not alter the physical properties.

NOTE The type and configuration of the mould depend on the holder of the brushing machine.

A.3.3.2 Enamel specimens

A.3.3.2.1 Selection

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

