
Dentistry — Products for external tooth bleaching

Médecine bucco-dentaire — Produits d'éclaircissement dentaire, à usage externe

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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.

ISO 28399 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 7, *Oral care products*.

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Introduction

Products for external tooth bleaching are used in dentistry for changing the colour of natural teeth towards a lighter or whiter shade. They are applied in the oral cavity directly on the outer surfaces of teeth. This International Standard includes requirements and test methods for products intended for external bleaching of natural teeth by chemical means.

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Dentistry — Products for external tooth bleaching

1 Scope

This International Standard specifies requirements and test methods for external tooth bleaching products. These products are intended for use in the oral cavity, either by professional application (in-office tooth bleaching products) or consumer application (professional or non-professional home use of tooth bleaching products), or both. It also specifies requirements for their packaging, labelling and instructions for use.

This International Standard is not applicable to tooth bleaching products:

- specified in ISO 11609;
- those intended to change colour perception of natural teeth by mechanical methods (e.g. stain removal) or using restorative approaches, such as veneers or crowns;
- auxiliary or supplementary materials (e.g. tray materials) and instruments or devices (e.g. lights) that are used in conjunction with the bleaching products.

This International Standard does not specify biological safety aspects of tooth bleaching products.

NOTE A tooth bleaching product can be evaluated for its biological safety using ISO 10993-1 and ISO 7405.

2 Normative references

The following referenced documents are indispensable for the application 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 1942:2009 *Dentistry — Vocabulary*

ISO 6344-1, *Coated abrasives — Grain size analysis — Part 1: Grain size distribution test*

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

3 Terms and definitions

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

3.1

bleaching

⟨natural teeth⟩ removal of intrinsic or acquired discolorations of natural teeth through the use of chemicals, sometimes in combination with the application of auxiliary means

NOTE Adapted from ISO 1942:2009, 2.28.

3.2 professional home use
(of a product) use prescribed by a professional and for use at home under the repeated supervision of the dentist

4 Classification

4.1 General

Products for external tooth bleaching can be classified for either:

- a) professional application; or
- b) consumer application.

NOTE Products for external tooth bleaching can be used alone or in conjunction with auxiliary means of application.

4.2 Products for professional application

These products are tooth bleaching products that are intended by the manufacturer to be applied only by dental professionals (in-office tooth bleaching products).

4.3 Products for consumer application

These products are tooth bleaching products that are intended by the manufacturer to be applied by the consumer (for professional home use or for non-professional home use).

NOTE Such external bleaching products can be prescribed by a dental professional or directly available to consumers.

5 Requirements

5.1 Concentration of active ingredients for bleaching

The concentration of active ingredients for bleaching (equivalent to hydrogen peroxide) delivered by the unexpired product according to manufacturer's instructions for use shall be within the range of +10 % and -30 % of the original concentration stated by the manufacturer for the unopened product, when tested in accordance with Annex A or other equivalent method.

5.2 Surface microhardness

The reduction in the Knoop hardness (KHN) or Vickers hardness (VHN) after the treatment shall be not more than 10 %, when tested in accordance with 6.3.

5.3 Surface erosion

Surface erosion of the teeth tested in accordance with B.6.1 shall be no more than three times the level which is caused by the positive control (B.6.2.1), when tested in accordance with Annex B or other equivalent methods.

6 Test methods

6.1 Preparation of tooth specimens

Prepare enamel and dentine specimens taken from a consistent location on extracted human or bovine teeth that have been stored in a 0,2 % solution (mass concentration) of sodium azide or other solutions of equivalent efficacy for disinfection purpose. Grind the specimen surface using a sequence starting at P400 and sequentially to a minimum of P1200 silicon carbide paper in accordance with ISO 6344-1 under a constant flow of water and then polish the surface using a slurry or paste of 0,3 µm mean particle size aluminium oxide. Ensure a minimum of 1 mm thickness of enamel or dentine tissue for the test specimen. Prevent dehydration of test specimens during the specimen preparation procedure.

6.2 Preparation and application of tooth bleaching product

The dispensing, processing and application of the tooth bleaching product used in tests shall follow manufacturer's instructions for use. The method of bleach application shall simulate the clinical procedure relative to quantity, frequency and duration of the application. Specimens shall be stored in a 37 °C artificial saliva solution similar to the one described in the ANSI/ADA Specification No. 41^[6] between bleaching intervals and for 24 h after the last bleach application prior to testing.

6.3 Surface microhardness

Evaluate enamel surface hardness before and after bleaching treatment.

Determine the surface microhardness with the KHN or VHN by applying a load of 0,49 N (equivalent to a 50 g load) for 15 s. Evaluate a minimum of 10 specimens for each group, with three indentations for each specimen. Prevent dehydration of test specimens during the specimen preparation procedure.

7 Packaging, marking and information to be supplied by the manufacturer

7.1 General

Additional information may be included at the discretion of the manufacturer or as required by regulation.

7.2 Packaging

The components of the material shall be supplied in properly sealed containers which adequately protect the contents and do not adversely affect the quality of the product.

7.3 Marking and instructions for use

For each package, the following applies.

- a) Information shall be clearly marked on the outermost package or containers appropriate to the product, as indicated in Table 1.
- b) Instructions shall accompany each package of the product and shall include the information appropriate to the product, as indicated in Table 1.

Table 1 — Requirements for marking and instructions for use

No.	Information	Outermost package	Container	Manufacturer's instructions for use
1	Name of the product	M	M	M
2	Identification or name of the manufacturer	M	M	M
3	Address of the manufacturer or the agent responsible for sale	M	—	M
4	Recommended conditions of storage	M	—	M
5	Manufacturer's lot or batch identification	M	M	—
6	Expiry date in accordance with ISO 8601 for the materials under the storage conditions recommended by the manufacturer	M	M	—
7	Classification of the materials (Clause 4)	M	—	M
8	Clinical application of the material (Clause 4)	—	—	M
9	Number of containers	M	—	—
10	Net mass of product in each container	M	M	M
11	Chemical name of active ingredient(s)	M	—	M
12	Concentration of active ingredient(s)	M	M	M
13	Concentration equivalent to hydrogen peroxide	M	M	M
14	Instructions for use	—	—	M
15	Recommended auxiliary device(s), exposure times and any special instructions for use of the equipment (for the materials requiring an auxiliary device only)	—	—	M
16	Specific contra-indication(s) and/or warning(s), such as "irritation", "avoid contact with eyes", as necessary	—	—	M
17	Statement equivalent to "It is recommended that you consult with your dental professional before using this product."	—	—	M
Explanation of symbols: "M" indicates mandatory information. "—" indicates non-mandatory information.				

Annex A (informative)

Test method for the measurement of hydrogen peroxide concentration

A.1 Principle

The content of hydrogen peroxide (H_2O_2) in tooth bleaching products is determined using a modified thiosulfate titration method.

A.2 Test condition

Perform the test at $(23 \pm 2)^\circ\text{C}$.

A.3 Procedure (modified thiosulfate titration method, USP^[10])

Equivalent methods can also be used.

Use analytic grade of sulfuric acid, potassium iodide, ammonium molybdate, sodium thiosulfate, starch and hydrogen peroxide. Conduct a titration calibration curve using a series of freshly prepared H_2O_2 solutions at concentrations that include the highest possible H_2O_2 concentration in the test product. Add approximately 1,0 g (weighing precision to 0,001 g) of test product or an amount appropriate to the test, with rapid stirring, to 400 ml distilled water that contains 10 ml of sulfuric acid (25 %), 25 ml potassium iodide (10 %), and 4 drops of ammonium molybdate solution (5 %). Use starch as the indicator, and perform the titration using 0,1 N (normality) sodium thiosulfate.

Determine the H_2O_2 content using the titration calibration curve.

When using standardized titrants (e.g. USP standard grade), construction of calibration curve is not necessary. Calculate the mass concentration of H_2O_2 from the following equation:

$$C = (1,701\ 18 \times V/m) \times 100$$

where

C is the mass concentration of H_2O_2 , expressed as a percentage;

V is the titre of 0,1 N sodium thiosulfate, in millilitres;

m is the mass of the test product dispensed, in grams.

Repeat the measurement five times ($n = 5$) and calculate the mean H_2O_2 concentration.

Annex B (informative)

Test method for the measurement of erosion of enamel and dentine caused by products for external tooth bleaching

B.1 Principle

The depth of erosion of enamel and dentine caused by products for external tooth bleaching is determined using a profilometer; see References [11] and [12].

B.2 Test condition

Perform the test at (23 ± 2) °C, i.e. ambient temperature.

NOTE When auxiliary devices, such as a light source specified as part of the application process of the product, is used in the test, the temperature of both the material and the tooth can increase depending on the device. See B.6.2 for test conditions for control solutions.

B.3 Standard reference erosion controls

B.3.1 Negative control

Use water of Grade 3 in accordance with ISO 3696 as the negative control.

B.3.2 Positive control

Use a 1,0 % solution (mass concentration) of citric acid (pH 3,9) as the positive control.

B.4 Apparatus

B.4.1 Contact profilometer¹⁾ or non-contact profilometer²⁾, with sensitivity $< 0,1 \mu\text{m}$.

B.4.2 Lapping and polishing machine, with sequential silicon carbide discs up to P1200 (according to ISO 6344-1).

Other methods of polishing enamel and dentine (e.g. diamond powder) that conform to the baseline requirements for specimens may be used.

1) Surfometer (Planar Products Ltd., Sunbury on Thames, UK) and Mitutoyo Surftest (SV-2000, Andover, UK) are examples of such an instrument. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

2) Proscan 2000 (Scantron Industrial Products Ltd., Monarch Centre, Venture Way, Taunton, UK) is an example of such an instrument. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

B.5 Specimen preparation

Use extracted human erupted or unerupted permanent teeth with the possible exception of lower incisors because of their small size, or extracted bovine teeth. Remove soft tissue remnants by scraping with a suitable instrument (e.g. curette, scalpel, etc.), and store the specimens in a 0,2 % solution (mass concentration) of sodium azide or other solutions of equivalent efficacy for at least 24 h prior to the specimen preparation. Section the tooth at the amelocemental junction with a dental bur or disc; use the coronal portion for enamel specimens and the radicular portion for dentine specimens. In the case of molars, section the crown vertically in bucco-lingual or mesio-distal directions to obtain buccal, lingual, mesial and distal slices of enamel. Use a diamond-edged circular cutting instrument to facilitate the procedure for slicing molar teeth. Similarly, section the root portion vertically in half or quarters so that an outer portion of root surface is available for polishing.

Place enamel and dentine portions outer face down in moulds measuring 25 mm × 25 mm × 3 mm and embed the specimen in epoxy resin for at least 24 h. Place specimens on the automatic lapping and polishing machine (B.4.2) with the outer face of enamel or dentine on the polishing discs. Initially expose the tissue surface with a coarse grit paper (e.g. P180 paper) and then final polishing sequentially using up to P1200 paper, which shall produce a flat surface with no more than an average profile of $\pm 0,3 \mu\text{m}$ measured by a contacting or non-contacting profilometer (B.4.1). Prevent dehydration of test specimens during the specimen preparation procedure.

Allocate at least six enamel and six dentine specimens to each product and the positive and negative control solutions. Prior to the erosion test, mask each specimen with two pieces of polyvinyl chloride (PVC) adhesive tape placed parallel to each other to expose a 2 mm to 3 mm wide window of enamel or dentine (see Figure B.1).

B.6 Erosion of enamel and dentine

B.6.1 Application of bleaching products

Apply the bleaching product to the specimens according to the manufacturer's instructions in terms of preparation of the formulation, application time and number of applications. If using an auxiliary device such as light/heat sources, follow the manufacturer's instructions.

B.6.2 Control treatments

B.6.2.1 Positive control treatment

Attach specimens to optically clear acrylic blocks using double-sided adhesive tape to prevent floating and then place the specimens in the 1,0 % solution of citric acid (pH 3,9) (B.3.2). Treat the specimens in 300 ml of citric acid solution (B.3.2) in a beaker placed in a water bath at 35 °C for 60 min. Stir the solution with the specimens by an overhead propelled stirrer at a constant speed of 270 r/min.

B.6.2.2 Negative control treatment

Treat the specimens in water of Grade 3 in accordance with ISO 3696 (see B.3.1) under the same conditions as for the positive control for 60 min.

B.7 Profilometer measurement method

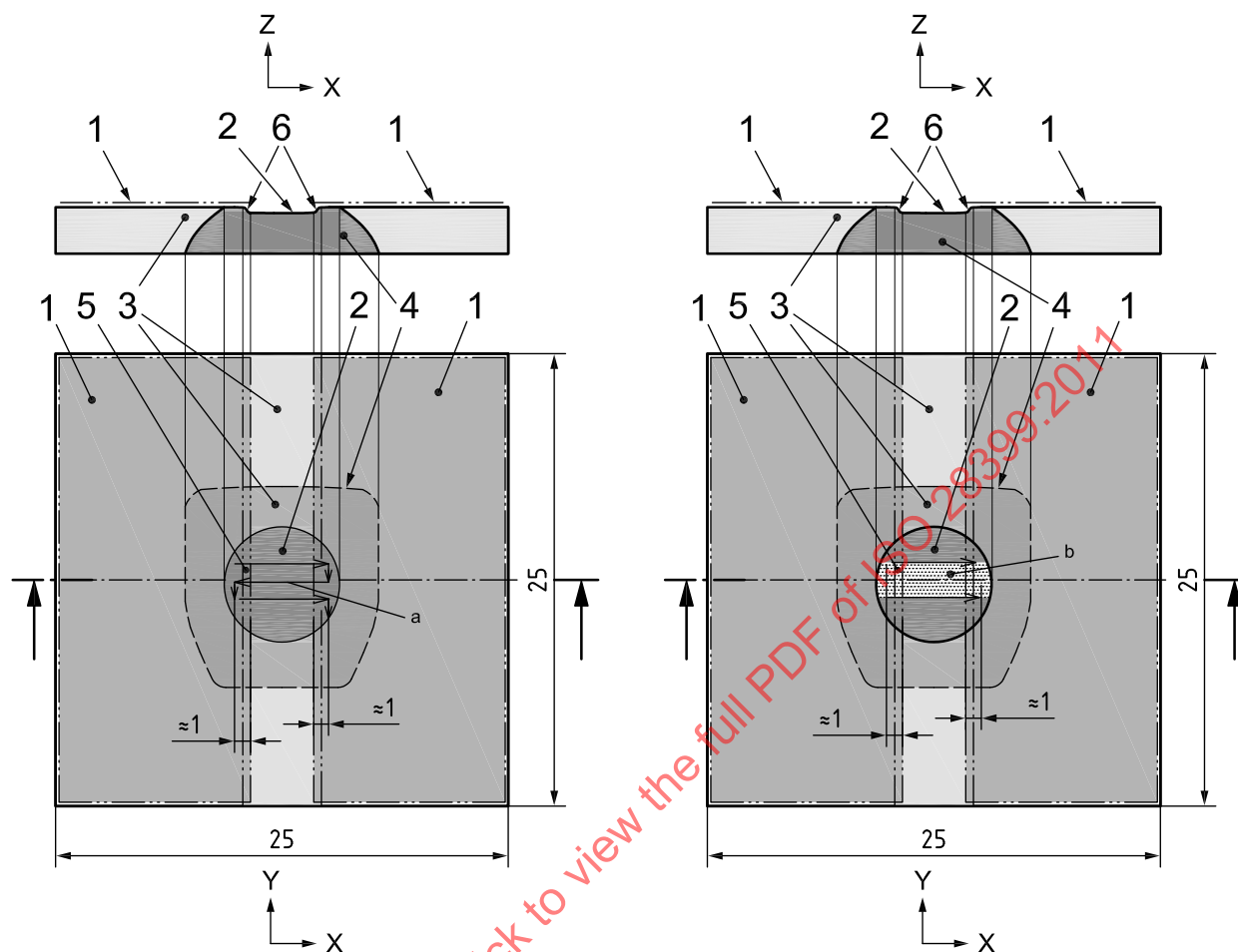
After treatments, remove the tape from specimens and measure the profile according to the operating method for the particular profilometers (see Figure B.1).

- **General measurement method for two-dimensional contact profilometers:** Measure the profile along the X-axis (see Figure B.1) from just inside the previously taped zone of the specimen (e.g. 1 mm) across the exposed zone, to just inside the opposite previously taped zone. Record the mean from at least 100 z -values across the scan as calculated by software. Conduct three scans from different points along the exposed zone and calculate the mean of the three readings.
- **General measurement method for three-dimensional contact and non-contact profilometers:** Measure the profile of the area containing both of the previously taped zones, such as 1 mm of length (in the X-axis) from each (left/right) edge of the exposed zone, and the treated zone of full length (in the X-axis, from the edge of the previously taped zone across the treated zone to the edge of the other previously taped zone).

Record all readings from these instruments in microns to the nearest two decimal places.

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Dimensions in millimetres



a) Profile measuring procedure for two-dimensional (2-D) profilometer

b) Profile measuring procedure for three-dimensional (3-D) profilometer

Key

- 1 zone previously covered by tape during treatment
- 2 prepared tooth surface exposed to treatment
- 3 cured resin block, in which a tooth specimen is moulded
- 4 tooth specimen
- 5 prepared tooth surface previously covered by tape during treatment
- 6 profile zone to be excluded from the measurement of the prepared tooth surface (because of partial erosion due to seepage under the tape during treatment)

a Illustration of a profile measurement of the zone exposed to treatment. The specimen is scanned along the X-axis for a linear scan using a 2-D profilometer (viewed from the top of the specimen). The depth of the profile of the treated surface is shown above along the Z-axis. Any z -values obtained in the area indicated by item 6 are excluded.

b Illustration of a profile measurement of the zone exposed to treatment over an area (x, y) scanned using a 3-D profilometer (viewed from the top of the specimen). The depth of the profile of the treated surface is shown above along the Z-axis. Any z -values obtained in the area indicated by item 6 are excluded.

Figure B.1 — Schematic illustration of specimen surface for profilometer measurement

Annex C (informative)

Test method for laboratory assessment of tooth bleaching efficacy

C.1 Principle

Preliminary data is obtained on tooth bleaching efficacy of a product using a laboratory method; see References [7], [8], [9], [13] and [14].

C.2 Materials, equipment and evaluation condition

C.2.1 Extracted human or bovine incisors

Prepare incisors in accordance with C.3.1 using appropriate infection control procedures.

C.2.2 Shade guide

Use an acceptable tooth shade guide under colour corrected lighting (e.g. 5 500 K) for visual assessment of tooth shade (see C.3.3.2).

C.2.3 Electronic colour measuring instrument

Use an acceptable instrument such as a colorimeter, spectrophotometer, or digital imaging device.

C.3 Procedures

C.3.1 Specimen preparation

Select extracted human incisors or bovine teeth of proper tooth colour [e.g. $\geq$ A2 or $\geq$ A3 of Vitapan Classical shade³⁾ (C.2.2), depending on the expected bleaching efficacy] with no labial lesions or restorations. Store the teeth in appropriate media, such as a 0,2 % solution (mass concentration) of sodium azide or other solutions of equivalent efficacy for disinfection purposes, ensuring that they have not been subject to any conditions that may alter their general properties. Clean the tooth surface of any stains and calculus. Assign a code number to each specimen, and assign the tooth specimens randomly into groups, with 6 to 10 specimens per group, depending on the baseline tooth colour and expected efficacy of the bleaching product. Prevent dehydration of test specimens during the specimen preparation procedure.

C.3.2 Bleaching treatment

Follow the manufacturer's instructions for bleaching. Prevent dehydration of test specimens during all procedures.

3) Vitapan Classical Shade Guide (Vita Zahnfabrik, Bad Säckingen, Germany) is one of such shade guides. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.