BS EN ISO 16408:2015

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Dentistry — Oral care products — Oral rinses

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National foreword

This British Standard is the UK implementation of EN ISO 16408:2015. It supersedes BS EN ISO 16408:2004 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CH/106/7, Oral hygiene products.

A list of organizations represented on this committee can be obtained on request to its secretary.

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European foreword

This document (EN ISO 16408:2015) has been prepared by Technical Committee ISO/TC 106 "Dentistry" in collaboration with Technical Committee CEN/TC 55 "Dentistry" the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 2016, and conflicting national standards shall be withdrawn at the latest by March 2016.

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The text of ISO 16408:2015 has been approved by CEN as EN ISO 16408:2015 without any modification.

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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.

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The committee responsible for this document is ISO/TC 106, *Dentistry*, Subcommittee SC 7, *Oral care products*.

This second edition cancels and replaces the first edition (ISO 16408:2004), which has been technically revised with the following changes:

— "heavy metals" has been replaced by the term *unintended heavy metals*, which has been defined in 3.3;

— reference to ISO 28888 and corresponding requirement in 5.1 were added;

— ambient storage conditions of real time test for determination of stability against ageing (7.4.1) was changed (23 ± 2) °C at (60 ± 15) % relative humidity;

— the bibliography was updated by including latest ISO/TC 217 standards.

Introduction

Oral rinses are used for oral hygiene purposes intended to provide health and/or cosmetic benefits.

This International Standard specifies the chemical and physical properties of oral rinses. Common labelling aspects are also specified in order to enhance international understanding and trade.

INTERNATIONAL STANDARD **ISO 16408:2015(E)**

Dentistry — Oral care products — Oral rinses

1 Scope

This International Standard specifies physical and chemical requirements and test methods for oral rinses. It also specifies the accompanying information such as the manufacturer's instructions for use, marking, and/or labelling requirements.

This International Standard is not applicable to other delivery systems (e.g. mouthsprays, foams, powders). It is not intended to describe regulatory aspects, e.g. methods of prescription.

This International Standard is not applicable to oral rinses available by prescription only.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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*

ISO 28888, *Dentistry — Screening method for erosion potential of oral rinses on dental hard tissues*

INCI, *International Nomenclature for Cosmetic Ingredients*

3 Terms and definitions

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

3.1
oral rinse
mouthrinse
mouthwash
liquid formulation used by the public for oral care purposes

[SOURCE: ISO 1942, 2009, 2.209]

3.2
mouthspray
liquid formulation in spray form for oral care purposes not requiring dilution with water

[SOURCE: ISO 1942, 2009, 2.185]

3.3
unintended heavy metals
heavy metal elements, which are detected in the analysis but not purposely included

4 Classification

Oral rinses shall be classified according to their application by the user as follows:

— **Type 1:** ready-for-use solutions;

— **Type 2:** concentrated solutions for use after dilution with water;

— **Type 3:** solutions for use after mixing.

5 Requirements

5.1 pH value

Oral rinses shall have a pH value between 3,0 and 10,5. If the pH value of an oral rinse is below 5,5 it shall pass a screening test as specified in ISO 28888.

Test the pH value in accordance with 7.1 and 7.3.

NOTE At the time of development of this International Standard, there is no evidence that oral rinses with pH values between 5,5 and 10,5 promote enamel erosion.

5.2 Total fluoride concentration and maximum amount of fluoride

The total fluoride concentration of one container of oral rinse of Type 1 shall not exceed a mass fraction of 0,15 %.

The maximum amount of ionic fluoride per single container shall not exceed 125 mg.

Test fluoride-containing oral rinses in accordance with Annex A.

As an alternative one of the procedures given in ISO 11609, Annex C,[3] or other validated method of similar sensitivity and accuracy, may be use, for example References [13] or [14].

5.3 Unintended heavy metals

The maximum total concentration of unintended heavy metals in oral rinses shall not exceed 20 mg/kg.

Test in accordance with a validated method, for example References [15], [16], [17] or [22].

If this is not suitable other method of similar sensitivity and accuracy shall be used.

NOTE There may be other potentially dangerous elements, especially arsenic, which are not covered by this International Standard as currently no analytical test methods and no effect levels are consented.

5.4 Compatibility with oral tissues

Oral rinses shall not cause irritation or damage to the oral hard and/or soft tissue, when used in accordance with the manufacturer's recommendation for frequency and duration of use and experience with known side effects.

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

5.5 Microbial contamination

The microbial contamination of oral rinses shall not exceed 100 colony-forming units (CFU) per gram. Oral rinses shall be free of pathogens.

Testing for microbial contamination shall be carried out according to a validated method, for example References [6], [7], [8], [9], [10], [11] or [12].

5.6 Stability against ageing

Oral rinses shall show no signs of deterioration, such as agglomeration or change in clarity, after being subjected to the determination of stability to ageing procedure specified in 7.4.

5.7 Container and/or dispensing system

The container and/or dispensing system shall neither contaminate nor permit contamination of the oral rinse inside such that it will affect its compliance with the requirements of Clause 5 after being subjected to the determination of stability to ageing described in 7.4.

5.8 Readily fermentable carbohydrates

Oral rinses 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.

6 Sampling

The oral rinses used for testing shall be representative of actual manufactured oral rinse and shall not be altered in any way.

Eight containers of oral rinses from the same manufacturing tracking code (e.g. batch code, lot number) shall be tested before the determination of stability to ageing (see 7.4).

7 Test methods

7.1 General

All tests shall be performed before and after the stability to ageing test (7.4).

7.2 Visual inspection

Before and after agitation, examine the oral rinse under a bright light with normal visual acuity without magnification.

7.3 Determination of pH value

Test the pH value of the oral rinse in its intended concentration for use.

Determine the pH value of the solution using a calibrated pH-meter with an accuracy of ±0,1 mV.

7.4 Determination of stability against ageing

7.4.1 Test

One of the following two tests shall be performed.

a) Accelerated test

   Store the oral rinse at (40 ± 2) °C for 3 months at (75 ± 5) % relative humidity or under such conditions of time and temperature as will simulate storage at room temperature for 30 months.

b) Real time test

Store the oral rinse at (23 ± 2) °C at (60 ± 15) % relative humidity for 30 months or for the period indicated by the expiry date listed on the product label [see 9.2 n)].

7.4.2 Compliance

Examine by visual inspection (7.2) of the oral rinse if requirement 5.6 is fulfilled.

7.5 Pass/fail criteria

Unless otherwise noted, if none of the samples fails, the oral rinse passes.

If one sample does not meet the minimum requirement, another eight samples shall be tested. If no more samples fail, the oral rinse passes. If a total of two or more samples of the 16 samples fail, the oral rinse fails.

8 Test report

The test report shall include at least the following information:

a) the name and address of the organization responsible for the test report;

b) a reference to this International Standard, i.e. ISO 16408;

c) the manufacturer's tracking code (e.g. batch code, lot number);

d) the test results and the method of determination used;

e) any unusual features noted during the determination;

f) if the oral rinse passed or failed the test.

9 Accompanying information

9.1 Manufacturer's instructions for use

The manufacturer's or supplier's instructions for use accompanying the oral rinse shall contain at least the following information:

a) information specified in 9.2, with the exception of d), f), and n), and, if necessary,

b) information on common side-effects,

c) recommended storage conditions (e.g. need for refrigeration).

9.2 Information on the primary container, and on the secondary container, if it exists

The following information, where appropriate, shall be given on the primary container, and also on the secondary container, if it exists:

a) the manufacturer's name and address and/or agent responsible in the country of sale;

b) trade name;

c) the wording "oral rinse" or equivalent, as defined in Clause 3;

d) the manufacturer's tracking code (e.g. batch code, lot number);

e) a list of ingredients:

   — a complete declaration according to the INCI-list (*International Nomenclature for Cosmetics Ingredients)* for cosmetic ingredients, if applicable,

   — a declaration according to the regional or national laws and/or national Pharmacopoeia, or

   — with descriptive names of ingredients.

   The identification of the ingredients shall be consistent with the guidelines, which states how the declaration should be made and the ingredients identified. This requirement is only applicable to the primary container, if there is no secondary container.

f) net volume, in millilitres;

g) if the oral rinse contains alcohol, the declaration of alcohol content, as volume fraction;

h) if the oral rinse contains fluoride, the concentration of fluoride, in milligrams per kilogram (mg/kg) or ppm (parts per million; 10^{-6}) by mass of fluoride ion;

i) instructions and warning for proper use with children;

j) the statement: Not suitable for children under 6 years of age unless medically recommended;

k) for oral rinses of Type 2, the statement: “Dilute according to the manufacturer’s instructions for use”;

l) for oral rinses of Type 3, the statement: “Mix according to the manufacturer’s instructions for use”;

m) the warning: “Do not swallow”;

n) if the shelf-life is less than 30 months, the expiry date for the oral rinse, expressed in accordance with ISO 8601, when stored under the manufacturer’s recommended storage conditions.

10 Packaging

Packaging should ensure the integrity of the contents of the container during storage and transportation. The packaging system for oral rinses is left to the discretion of the manufacturer.

Annex A
(normative)

Determination of fluoride in oral rinses containing ionic fluoride compounds

A.1 Principle

This test method is used for the determination of fluoride in oral rinses containing ionic fluoride compounds.

This test is a type test.

NOTE At the time of development of this International Standard, standardization work on a harmonized standardised test method for oral fluoride analysis has started.

A.2 Reagents and/or materials

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade.

A.2.1 Deionized water, in accordance with ISO 3696, grade 2.

A.2.2 Fluoride standard solution, commercially available or prepared with sodium fluoride (NaF).

A.2.3 Total Ionic Strength Adjustment Buffer (TISAB) solution, with cyclohexanediamine tetraacetate (CDTA). Other buffer solutions such as ammonium acetate buffer, applicable to fluoride analysis, may also be used.

Ammonium acetate buffer (pH 5,3) is prepared by dispersing of 16 g of ammonium chloride, 23 g of ammonium acetate and 0,4 g of *trans*-1,2-cyclohexanediamine-*N,N,N',N'*-tetraacetate monohydrate in about 80 ml of water and by dissolving this solution after mixing and heating. The pH value of this buffer is adjusted to 5,3 with acetic acid, and the buffer is diluted with deionized water to 100 ml.

A.3 Apparatus

The following apparatus shall be used.

A.3.1 Laboratory balance, with a reading accuracy of 0,01 g.

A.3.2 Flask, of capacity 20 ml.

A.3.3 Fluoride ion selective electrode (F-ISE), with reference electrode, or combination F-ISE/reference electrode pair.

A.3.4 Graduated cylinder, of capacity 15 ml to 50 ml.

A.3.5 Magnetic stirring apparatus, with PTFE-coated magnetic stirring bar and magnetic stir plate.

A.3.6 pH/mV-electrometer (pH meter), with an accuracy of ±0,05 pH units (±0,1 mV), calibrated.

A.3.7 Pipette, of capacity (1,0 ± 0,1) ml.

A.3.8 Plastic vial, or any small beaker or container, 10 ml or more capacity.

A.3.9 Washing bottle.

A.4 Preparation of solutions and calibration curve

A.4.1 Preparation of standard solution for calibration

Make successive dilutions of the fluoride standard solution (A.2.2) to obtain a set of working standards which includes 5 mg/kg, 10 mg/kg (10^{-5} mol/l), 50 mg/kg, 100 mg/kg (10^{-4} mol/l), and 150 mg/kg of fluoride.

A.4.2 Preparation of calibration curve

Use the following procedure to prepare the calibration curve.

a) Pipette 1,0 ml of each standard solution (A.4.1) into a plastic vial (A.3.8).

b) Add 1,0 ml of TISAB solution (A.2.3), and add a magnetic stirring bar (A.3.5) to each plastic vial. Mix thoroughly.

c) Insert the fluoride ion selective electrode (A.3.3) and reference electrodes into the liquid in the plastic vial containing the first standard solution. Make sure no air bubbles have been trapped under the electrode.

d) Record the millivolt reading to 0,1 mV at the steady potential difference with the mV electrometer (A.3.6).

e) Conduct at least two measurements for millivolt readings, until the difference between the two millivolt readings is less than 0,2 mV.

f) Repeat steps c) to e) for each of the other standard solutions.

g) Construct a calibration curve of millivolts versus the log of the fluoride ion concentration of the standard, expressed in milligrams per kilogram.

NOTE The slope of the calibration curve should be linear.

A.4.3 Preparation of sample solution

In duplicate, pipette (1,0 ± 0,1) g of each sample into a 20 ml flask (A.3.2).

Add (9,0 ± 0,1) ml of deionized water (A.2.1) to the flask and mix thoroughly. This is the sample solution.

A.5 Sample analysis

Determine the fluoride ion concentration in the sample solution as follows.

a) Pipette accurately an equal amount of sample solution (A.4.3) and TISAB solution (A.2.3) into a plastic vial (A.3.8) and mix thoroughly.

b) Insert the fluoride ion selective electrode (A.3.3) and reference electrodes into liquid that contains the sample and buffer solution, in the plastic vial (A.3.8). Make sure no air bubbles are trapped under the electrode.

c) Record the millivolt reading to the nearest 0,1 mV at the steady potential difference with the mV electrometer.

d) Use the calibration curve of standard solutions to determine the fluoride ion concentration in the sample solution, in milligrams per kilogram.

A.6 Expression of results

A.6.1 Expression

The fluoride ion concentration shall be expressed in milligrams per kilogram of the oral rinse solution unless otherwise required.

NOTE Regulatory requirements in some regions require the expression of fluoride ion concentration in ppm = parts per million (10^{-6}).

A.6.2 Calculation of fluoride ion concentration

Calculate the fluoride ion concentration of one container of oral rinse using Formula (A.1) (units see A.6.1):

$$c_{OR} = c_S * 10 \quad \text{(A.1)}$$

where

c_{OR} is the fluoride ion concentration of oral rinse, in milligram per kilogram (10^{-6});

c_S is the fluoride ion concentration of sample solution, in milligram per kilogram (10^{-6}).

A.6.3 Calculation of mass of ionic fluoride

Calculate the mass of ionic fluoride per single container of oral rinse using Formula (A.2) (units see A.6.1):

$$m_{OR} = c_{OR} * m \quad \text{(A.2)}$$

where

m_{OR} is the mass of ionic fluoride of oral rinse, in milligram;

c_{OR} is the fluoride ion concentration of oral rinse, in milligram per kilogram (10^{-6});

m is the mass of the solution in one container of oral rinse, in kilogram.

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