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Dentistry — Screening method for erosion potential of oral rinses on dental hard tissues

Médecine bucco-dentaire — Méthode de criblage de l'érosion potentielle des tissus durs dentaires due aux rinçages oraux

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Foreword

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

Introduction

This International Standard describes a screening method for assessing the erosion potential of dental hard tissues associated with the use of oral rinses.

The primary aim of this International Standard is to provide methodology for screening oral rinses for the potential for tooth erosion.

Oral rinses should not cause adverse reactions to the oral soft and hard tissues when used in accordance with the manufacturer's recommendation for frequency and duration of use.

The range of known side effects and biological hazards is wide and complex. The tissue interaction with a constituent material alone cannot be considered in isolation from the overall device design. Thus, in designing an oral rinse, the choice of the best material with respect to its tissue interaction might result in a less functional product, tissue interaction being only one of a number of characteristics to be considered in making that choice. Where a material is intended to interact with tissue in order to perform its function, the biological response to this interaction can be evaluated.

Dentistry — Screening method for erosion potential of oral rinses on dental hard tissues

1 Scope

This International Standard specifies a screening method for the erosion potential of non-fluoridated oral rinses on dental hard tissues.

The results of the screening method are intended for use in enamel and/or dentine erosion models.

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 78-2, *Chemistry — Layouts for standards — Part 2: Methods of chemical analysis*

ISO 1942, *Dentistry — Vocabulary*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 78-2, ISO 1942 and the following apply.

3.1
dental erosion
progressive loss of calcified dental hard tissue by chemical processes that do not involve bacterial action

[SOURCE: ISO 1942:2009, 2.292]

4 Test method

4.1 General

The risk of enamel and dentine erosion due to oral rinses shall be assessed.

This method is intended to provide initial screening of potential for erosion for all non-fluoridated oral rinses.

In case a product fails the screening test, test methods that are more complex and close to clinical conditions shall be applied.

4.2 Maximum decrease in pH

The maximum allowable decrease in pH of this test method shall be 1,0.

Should a decrease of the pH greater than 1,0 be determined, then the oral rinse fails this screening test. In this case, test methods that are more complex and close to clinical conditions shall be performed in order to establish the erosive capacity of the oral rinse as specified in ISO 16408.

4.3 Reagents

4.3.1 Calcium chloride dihydrate ($CaCl_2$ 2·H_2O), reagent grade.

4.3.2 Citric acid ($C_6H_8O_7$), reagent grade.

4.3.3 Potassium hydroxide (KOH), reagent grade.

4.3.4 Hydrochloric acid (HCl), reagent grade.

4.3.5 Sodium azide (NaN_3), reagent grade.

4.3.6 Trisodium citrate dihydrate ($C_6H_5Na_3O_7$ 2·H_2O), reagent grade.

4.3.7 Potassium dihydrogen phosphate (KH_2PO_4), reagent grade.

4.3.8 Distilled water (H_2O), complying with grade 2 of ISO 3696.

4.3.9 Reference citrate buffer solutions.

Prepare three reference citrate buffer solutions according to 4.6.2.

— Solution 1: 1,0 % citric acid at pH 3,60 at 25 °C;

— Solution 2: 0,25 % citric acid at pH 3,68 at 25 °C;

— Solution 3: 0,07 % citric acid at pH 3,77 at 25 °C.

4.4 Apparatus

4.4.1 One 50 ml vessel, composed of (borosilicate) glass.

4.4.2 Analytical balance, with an accuracy of 0,1 mg or better.

4.4.3 Magnetic stirring apparatus, with PTFE-coated magnetic stirring bar.

4.4.4 Volumetric flask, 1 l.

4.4.5 Beaker, 100 ml, made of borosilicate glass, clean.

4.4.6 Pipette, capable of measuring 1 ml to 0,01 ml.

4.4.7 Thermometer, with an accuracy of 0,1 °C or better.

4.4.8 pH meter (pH electrode), with a sensitivity of ±0,05 pH units, calibrated.

EXAMPLE 1 Example for calibration: use pH 2,0, 4,0, and 6,0 standards or pH 1,68, 4,01 and 6,86 at 25 °C. Use pH standard solutions prepared in accordance with appropriate ISO Guides. Check for a linear response with a slope of at least 58 mV per pH unit.

NOTE Ready-to-use pH standards can be used.

4.5 Sampling

Use two representative samples from each of three batches of the oral rinse (total: six samples).

4.6 Test method

4.6.1 Preparation of screening solutions

4.6.1.1 Preparation of stock solutions

Prepare the following two stock solutions.

— Stock solution A: 1 mol/l $CaCl_2$: 147,01 g $CaCl_2 \cdot 2H_2O$ in 1 l distilled H_2O add 0,02 g NaN_3.

— Stock solution B: 1 mol/l KH_2PO_4: 136,09 g KH_2PO_4 in 1 l distilled H_2O add 0,02 g NaN_3.

NOTE The NaN_3 is added as a preservative to prevent microbial contamination while in storage. Should growth be observed, it is intended that new stock solutions be prepared.

4.6.1.2 Preparation of diluted screening solution

Prepare diluted screening solution (calcium phosphate) as follows.

To 500 ml distilled water (4.3.8) in a 1 l volumetric flask, add 1,266 ml 1 mol/l $CaCl_2$ (Stock A) and 0,760 ml 1 mol/l KH_2PO_4 (Stock B).

Adjust to pH = (5,05 ± 0,05) with HCl.

NOTE 1 KOH can be required to raise the pH; typically, HCl is need to lower the pH to the appropriate value.

Dilute to 1 l with distilled water (4.3.8).

NOTE 2 This solution is prepared fresh daily from the stock calcium phosphate solutions.

4.6.2 Preparation of reference buffer citrate solutions

Prepare the reference buffer solutions as described in Table 1 in clean 100 ml glass beakers or other suitable container.

Weigh powdered anhydrous citric acid and trisodium citrate dihydrate on separate weighing dishes; combine crystals in a 100 ml volumetric flask and add distilled water (4.3.8) until the meniscus nears the graduation line.

Determine the pH of these solutions using a suitably calibrated pH electrode and meter, while agitating using a magnetic stirrer.

If the pH is more than ±0,05 units away from the expected pH, adjust this pH accordingly with 0,1 mol/l potassium hydroxide solution or 0,1 mol/l hydrochloric acid to the expected value.

Add distilled water (4.3.8) to make up to the final volume of 100 ml.

Table 1 — Preparation of reference buffer citrate solutions

Solution number	Citric acid mass fraction, %	Trisodium citrate dihydrate ($C_6H_5Na_3O_7 \cdot 2 \cdot H_2O$) mass	Citric acid ($C_6H_8O_7$) mass	Expected pH
1	1,00	0,451 g	0,705 g	3,60
2	0,25	0,114 g	0,178 g	3,68
3	0,07	0,031 g	0,048 g	3,77

4.6.3 Screening procedure

4.6.3.1 Test procedure

Perform the test at an ambient temperature of between 18 °C and 27 °C.

Measure 25 ml of diluted screening solution (calcium phosphate solution) into the 50 ml reaction vessel. Record the temperature of the solution. Stir at a moderately fast rate (if controllable, set above 100 r/min) and hold this rate constant throughout the experiment. Place a calibrated pH electrode in the solution and monitor the pH until a constant pH is determined.

4.6.3.2 Evaluation

Add 250 µl (0,25 ml) of the test material (i.e. reference buffer or oral rinse product) into the stirring solution.

4.6.3.3 Test duration

The reaction will be terminated after recording steady pH.

4.6.3.4 Test replication

Repeat test four times with each test sample.

4.6.3.5 Data recording and treatment

For each test, record pH of test material (buffer or oral rinse), starting pH of calcium phosphate test solution, pH of test solution after addition of test material, and pH change (starting pH minus final pH).

Report raw data for each test material.

4.6.3.6 Evaluation

Calculate the mean pH change out of the four repetitions for each of the six samples and standard deviations of pH change for the test materials. If the mean pH reduction for each of the six samples is equal to or less than 1,0, the oral rinse passes the test. If one sample does not meet the requirement, the rinse shall be tested for erosion by other appropriate methods as specified in ISO 16408.

5 Test report

A test report shall be prepared on the test procedure. The test report shall contain the following information:

a) complete identification of the tested oral rinse, including name of the product, manufacturer, lot number, type of administration (e.g. can, paste, syringe);

b) storage conditions of the oral rinse;

c) number of samples tested;

d) indication of pass or fail;

e) deviations from the test as described in this International Standard, if appropriate;

f) reference to this International Standard, i.e. ISO 28888;

g) date of test;

h) date and signature of the person carrying out the test.

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