INTERNATIONAL STANDARD

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Dentistry — Fluoride varnishes

Médecine bucco-dentaire — Vernis fluorés

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Foreword

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The committee responsible for this document is ISO/TC 106, *Dentistry,* Subcommittee SC7, *Oral care products*.

Introduction

Fluoride varnishes are used in dentistry primarily for caries prevention and reduction of dentine hypersensitivity. Fluoride varnishes are applied in the oral cavity directly on the outer surfaces of teeth and fillings, and are intended to remain at least for several hours.

Specific qualitative and quantitative test methods for demonstrating freedom from unacceptable biological hazard are not included in this International Standard, but it is recommended that, for the assessment of possible biological or toxicological hazards, reference should be made to ISO 10993-1 and ISO 7405.

Dentistry — Fluoride varnishes

1 Scope

This International Standard specifies requirements and their test methods for total digestible fluoride content in dental varnishes containing fluoride, intended for use in the oral cavity directly on the outer surfaces of teeth and fillings. It also specifies the requirements for their packaging and labelling, including the instructions for use. This International Standard covers fluoride varnishes to be applied by dental health care workers.

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*

3 Terms and Definitions

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

3.1
consumer packaging
retail packaging
sales packaging
package, with labelling and contents as a sales unit to the user or consumer at the point of retail

Note 1 to entry: A consumer packaging can consist of a secondary packaging wrapping or container covering one or more primary packaging elements or, it can consist of a single primary packaging element containing the product.

Note 2 to entry: Adapted from ISO 21067:2007, definition 2.2.5.

3.2
fluoride varnish
dental product, in paintable liquid form containing fluoride compound for topical application to the outer surfaces of teeth and fillings, intended primarily to prevent tooth caries or reduce dentine hypersensitivity

3.3
total fluoride
mass percent of fluoride in the fluoride varnish

4 Requirements

4.1 Total fluoride content

The total fluoride content shall not deviate by more than 20 % from the stated amount on the package when tested in accordance with 6.1 or other validated methods for total fluoride content.

5 Sampling

Use packages prepared for retail sale from the same batch containing enough material to carry out the specified tests, plus an allowance for repeat tests, if necessary.

6 Test method

6.1 Test for total fluoride content

6.1.1 Chemicals and solutions

6.1.1.1 Total ionic strength adjusting buffer (TISAB) solution, described in ASTM D1179-10:2010 18.1 or alternative.

NOTE TISAB II: Sodium chloride 5,8 % (w/v), acetic acid 5,7 % (w/v), CDTA 0,4 % (w/v) adjust to pH 5 to pH 5,5 with sodium hydroxide (5 mol/L). Examples are TISAB II® (Orion Research). An acceptable product (TISAB II®) is available from Thermo Fisher Scientific, Beverly, MA 01915 USA. This information is given only for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

6.1.1.2 50 % Diluted TISAB solution, dilute TISAB with deionized water 1:1 by volume.

6.1.1.3 Fluoride standard solution, commercially available or prepared with sodium fluoride at 0,10 mol/L.

6.1.1.4 Chloroform, reagent grade.

6.1.1.5 Deionized water, in accordance with ISO 3696:1987, grade 2.

6.1.1.6 Potassium hydroxide at 1 mol/L.

6.1.2 Apparatus

6.1.2.1 Chloroform resistant vials and caps, 25 mL or more capacity.

6.1.2.2 Balance accurate to 0,000 1 g.

6.1.2.3 Magnetic stir bar.

6.1.2.4 Magnetic stir plate.

6.1.2.5 Pipette, capable of measuring 1 mL to 0,01 mL.

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

6.1.2.7 Plastic vial (or beaker or small container), 25 mL or more capacity.

6.1.2.8 pH/mV electrometer (pH meter), with a sensitivity of ± 0,1 mV.

Perform tests on “as received” samples.

6.2 Test conditions

Perform tests at room temperature with no more than 2 °C variation.

6.2.1 Preparation of standard solution

Make successive dilutions of the fluoride standard solution to obtain a set of working standard solutions that are 10^{-2}, 10^{-3}, 10^{-4}, 10^{-5} mol/L NaF.

6.2.2 Preparation of calibration curve

a) Pipette 5,0 mL of each standard solution into a separate plastic vial (or beaker or small container).

b) Add 5,0 mL of TISAB solution, add a magnetic stir bar to each plastic vial. Mix thoroughly.

c) Insert the fluoride ion selective and reference electrodes into the liquid in the plastic vial containing the first standard solution.

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

e) Conduct at least two measurements for millivolt readings until the difference between the two 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.

NOTE The slope of the calibration curve should be linear and no less than 55 mV per decade ($-\log_{10}$ [F]), and the l c coefficient of determination (r^2) should be greater than 0,98.

6.2.3 Sample preparation

a) Place open chloroform resistant vial that can be capped, such as a scintillation vial, on balance and tare to zero.

b) Dispense 1 to 3 drops (0,05 g to 0,15 g) of varnish product onto the bottom of the container.

c) Determine and record the mass of the amount of varnish [$\text{Varnish}_{\text{mass}}$] added to ± 0,000 1 g.

d) If the varnish sample contains fluorosilane, add 1,0 mL of 1 mol/L KOH to the varnish in the container and mix for 5 min. If the varnish sample does not contain fluorosilane then skip this step.

e) Add (4 ± 0,5) mL of chloroform into the container and stir.

f) Add (10,0 ± 0,1) mL of TISAB buffer to the chloroform + sample.

g) Cap and vigorously shake the mixture to dissolve the resin into the chloroform and all the fluoride into the TISAB. Be careful to allow the pressure in the vial to escape by loosening the cap briefly and retightening.

h) Add magnetic stir bar.

i) Stir for at least 1,5 h at room temperature.

NOTE In some cases the TISAB/Chloroform/Varnish mixture does not mix well with magnetic stirring, it may be necessary to place the sample vial into a rotator or shaker to achieve better mixing.

j) Turn off magnetic stir plate for at least 2 h (to allow the chloroform and the TISAB to separate);

k) If the top (aqueous) layer is not uniform, gently homogenize (mix) just that layer prior to sampling for further dilution.

6.2.4 Sample Analysis

a) Pipette 1,0 mL of aqueous (TISAB) layer (prevent to include parts of the lower chloroform layer) into a 25 mL container.

b) Pipette 1,0 mL deionized water into the same container.

c) Add 18,0 mL 50 % diluted TISAB solution to the container and stir.

d) Insert the fluoride ion selective and reference electrodes into the sample liquid in the vial. Record the millivolt reading to the nearest 0,1 mV at the steady potential difference.

e) Use the calibration curve of standard solutions to determine the fluoride ion concentration in the sample solution in mol/L.

6.2.5 Calculations

a) Determine the apparent concentration [F_{dil}] (mol/L) of the diluted sample from the standard curve.

b) Multiply the [F_{dil}] by 100 and divide by [$Varnish_{mass}$] = [F] mmol/g product.

NOTE When calculating the [F] mol/L of the standard, it is common practice to ignore the volume fraction of 50 % dilution caused by the addition of TISAB because the standards and samples all receive the same dilution. Should the standard curve be calculated with the actual [F] mol/L (including the volume fraction of 50 % TISAB dilution) then the dilution factor to be used in the calculations 6.2.5.b) will be 200 in place of 100 when calculating the [F] mmol/g product.

c) Multiply [F] mmol/g product by 19 000 = [mg F/g].

d) Multiply [F] mmol/g product by 41 990 = [mg NaF/g].

e) Divide [mg F/g] by 10 000 = [mass % F].

f) Divide [mg NaF/g] by 10 000 = [mass % NaF].

7 Test report

The test report shall include at least the following information:

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

b) a reference to this International Standard (i.e. ISO 17730);

c) the manufacturer's tracking code;

d) the test methods used and their results;

e) any unusual features noted during the tests;

f) whether the fluoride varnish passed or failed the test.

8 Instructions for use

Instructions shall indicate the method of application of the fluoride varnish to teeth.

9 Packaging, labelling, and marking

9.1 Packaging

The materials shall be supplied in containers or capsules that afford adequate protection and have no adverse effect on the quality of the contents.

9.2 Labelling

Each container shall be marked with at least the following information:

a) the manufacturer's name and address and/or the authorized representative;

b) the trade name of the material;

c) the type of fluoride (for example: sodium fluoride);

d) the total fluoride content (for example: micrograms per gram fluoride or mass % fluoride);

e) the manufacturer's tracking code;

f) recommended conditions of storage;

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

h) the expiry date, expressed in accordance with ISO 8601, for the material if stored under the manufacturer's recommended conditions;

i) any hazard warnings (flammable, etc.).

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