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Dentistry — Fluoride varnishes

Médecine bucco-dentaire — Vernis fluorés

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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*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 55, *Dentistry*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 17730:2014), which has been technically revised.

The main changes compared to the previous edition are as follows:

- Additional subclause to indicate three samples are tested for total fluoride. Two aliquots are analysed for each sample.
- A requirement of minimum soluble fluoride release potential and a relevant test method have been added.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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 hazards are not included in this document, 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.

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Dentistry — Fluoride varnishes

1 Scope

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

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 648:2008, *Laboratory glassware — Single-volume pipettes*

ISO 835:2007, *Laboratory glassware — Graduated pipettes*

ISO 1942, *Dentistry — Vocabulary*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 8601-1, *Date and time — Representations for information interchange — Part 1: Basic rules*

ISO 8601-2, *Date and time — Representations for information interchange — Part 2: Extensions*

ISO 19448, *Dentistry — Analysis of fluoride concentration in aqueous solutions by use of fluoride ion-selective electrode*

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:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1

fluoride varnish

dental product, in paintable liquid/paste 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

3.2

total fluoride content

mass percent of fluoride in the *fluoride varnish* (3.1)

3.3

fluoride release potential

detectable soluble fluoride released from the *fluoride varnish* (3.1) into an aqueous medium

3.4

outermost packaging

packaging used to combine material and additional items, including instructions for use and any proportioning or mixing devices that are supplied with the material

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 [6.2](#) or other validated methods for total fluoride content.

4.2 Fluoride release potential

The fluoride release potential shall be equal to or higher than 14,2 ng F/mm² as determined by the test method in [Annex A](#). Other validated measurement methods on fluoride release potential with similar sensitivity and accuracy to [Annex A](#), conforming to practices and principles found in References [\[1\]](#) to [\[6\]](#) may be used.

NOTE Quantitative measurement using method in [Annex A](#) is not established to provide links to clinical findings.

5 Sampling

Use packaged material(s) of fluoride varnishes 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. Perform tests on the materials as received.

6 Measurement and test methods

6.1 Test conditions

Perform tests at 23°C ± 2 °C.

6.2 Test for total fluoride content

6.2.1 Chemicals and solutions

6.2.1.1 Total ionic strength adjustment buffer (TISAB) solution, described in ASTM D1179-16: 2016; see Reference [\[8\]](#) or alternative.

NOTE 1 TISAB¹⁾: Dissolve sodium chloride 5,8 g, acetic acid 5,7 g, CDTA 0,4 g in 90 ml of deionized water. Adjust to pH 5,5 with sodium hydroxide (5 mol/l) and make up to a total of 100 ml solution with deionized water.

NOTE 2 It is known that interfering ions of, for example, calcium, aluminium, iron, magnesium, sulphate and phosphate salts can affect the result unpredictably; see References [\[9\]](#) and [\[10\]](#). The use of TISAB III® or TISAB IV® buffer can be considered.

6.2.1.2 50 % Diluted TISAB solution, dilute TISAB with deionized water 1:1 by volume.

1) Examples are TISAB II® (Orion Research). Acceptable products are TISAB II®, TISAB III®, and TISAB IV® available from Thermo Fisher Scientific, Waltham, MA 02451 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.

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

6.2.1.4 Chloroform, reagent grade.

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

6.2.1.6 Potassium hydroxide at 1 mol/l.

6.2.2 Apparatus

6.2.2.1 Chloroform resistant vials and caps, 25 ml or more capacity.

6.2.2.2 Balance, accurate to 0,000 1 g.

6.2.2.3 Magnetic stir bar.

6.2.2.4 Magnetic stir plate.

6.2.2.5 Pipette, of nominal capacity 1,0 ml to smallest scale division 0,01 ml, 10,0 ml to 0,02 ml, and 20,0 ml to 0,03 ml, in accordance with ISO 835:2007, Class A.

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

6.2.2.7 Plastic small container (e.g. vial or beaker), 25 ml or more capacity capable of allowing stirring with a stir bar operated by a stirrer.

6.2.2.8 pH/mV electrometer (pH meter), with a sensitivity of $\pm 0,1$ mV.

6.2.3 Procedure

6.2.3.1 Preparation of standard solutions

Prepare standard solutions in accordance with ISO 19448.

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.

6.2.3.2 Preparation of calibration curve

Prepare calibration curve in accordance with ISO 19448.

6.2.3.3 Sample preparation

- Place open chloroform resistant small container that can be capped, such as a scintillation vial, on balance and tare to zero.
- Dispense 0,05 g to 0,15 g of varnish product onto the bottom of the small container.
- Determine and record the mass of the amount of varnish [m_{varnish}] accurate to 0,000 1 g.
- If manufacturer's instruction for use indicates that the varnish contains fluorosilane, add 1,0 ml of 1 mol/l KOH to the varnish in the small container. Cap and vigorously swirl the small container for 5 min to mix the sample with the KOH. If the varnish does not contain fluorosilane then skip this step.

- e) Add magnetic stir bar.
- f) Add $(4,0 \pm 0,5)$ ml of chloroform into the small container and stir.
- g) Add $(10,0 \pm 0,1)$ ml of TISAB to the mixture (chloroform + sample) in [6.2.3.3 f\)](#).
- h) Cap and vigorously shake the mixture to dissolve the varnish product 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.
- i) Stir for at least 1,5 h at room temperature.

NOTE In case the TISAB/Chloroform/Varnish mixture does not mix well with magnetic stirring, it can be necessary to place the small container containing the mixture 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 (i.e. mix) just that layer prior to sampling for further dilution.
- l) Prepare two more samples for a total of three samples.

6.2.3.4 Sample analysis

- a) If the varnish product does not contain fluorosilane then pipette 1,0 ml of aqueous (TISAB) layer (prevent including parts of the lower chloroform layer) into a 25 ml container.

If the varnish product contains fluorosilane then pipette 1,1 ml of the aqueous layer (TISAB and KOH) into a 25 ml container. Take care to prevent including parts of the lower chloroform layer.

- b) If the varnish product does not contain fluorosilane then pipette 1,0 ml deionized water into the same container.

If the varnish product contains fluorosilane then pipette 0,9 ml deionized water into the same container.

- c) Add 18,0 ml of 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 concentration of fluoride ion in the sample solution in mol/l.
- f) Repeat analysis on a second aliquot from the same sample.
- g) Conduct analysis on the second and third samples in accordance with [6.2.3.4 a\) to f\)](#) for a total of 6 analyses.

6.2.3.5 Calculations

- a) Determine the apparent concentration $[F_{dil}]$ (mol/l) of the diluted sample from the standard curve.
- b) Multiply the value of fluoride concentration ($[F_{dil}]$) by 100 and divide by the value of mass of varnish sample ($[m_{varnish}]$) to obtain $[F]$ (mmol F/g) product.

NOTE 1 When calculating $[F]$ (mmol F/g) product from $[F_{dil}]$ (mol/l) the multiplication factor of 1 000 mmol per mol is divided by 10 the dilution factor of the analysis prior to division by the varnish sample mass.

NOTE 2 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 varnish 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 [6.2.3.5 b](#)) will be 200 in place of 100 when calculating the [F] (mmol F/g) product.

- c) Multiply [F] (mmol/g) by 19 to obtain [F] (mg/g).
- d) Multiply [NaF] (mmol/g) by 42 to obtain [NaF] (mg/g).
- e) Divide [F] (mg/g) by 10 to obtain [F] (mass %).
- f) Divide [NaF] (mg/g) by 10 to obtain [NaF] (mass %).

6.2.3.6 Interpretation of results

Calculate the mean value and standard deviation of the six analyses in accordance with [6.2.3.5](#).

Check the results for conformity with the requirement in [4.1](#).

6.3 Tests for conformance with “Instructions for use” and “Packaging and labelling”

Visual inspection of fluoride varnishes packages shall be performed in accordance with [Clause 8](#) and [Clause 9](#).

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 document and the date of the document (i.e. ISO 17730:2020);
- c) the manufacturer's tracking code for the varnish product tested;
- d) the date of the tests
- e) the test methods used;
- f) any unusual features noted during the tests;
- g) whether the fluoride varnish passed or failed the requirements.

8 Instructions for use

Instructions shall indicate the method of application of the fluoride varnish to teeth as listed in [Table 1](#).

9 Packaging and marking/labelling

9.1 Packaging

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

9.2 Marking/labelling

Requirements for marking/labelling are listed in [Table 1](#).

Table 1 — Requirements for marking and instruction for use

No.	Information	Outermost Packaging	Container	Single dose packaging	Manufacturer's instructions for use
1	Name of the product	M	M	M	M
2	Identification or name of manufacturer	M	M	—	M
3	Address of the manufacturer or the agent responsible for sale	M	—	—	M
4	Recommended condition of storage	M	—	—	M
5	Manufacturer's lot or batch identification	M	M	M	—
6	Expiry date in accordance with ISO 8601-1 and ISO 8601-2 for the materials under the storage condition recommended by the manufacturer	M	M	M	—
7	Clinical application of the material	—	—	—	M
8	Number of containers	M	—	—	—
9	Net mass in grams, or net volume in millilitres	M	M	—	—
10	The type of fluoride (for example: sodium fluoride)	M	M	—	M
11	The total fluoride content (for example: Micrograms fluoride per gram varnish or mass % fluoride)	M	M	—	M
12	Instructions for use	—	—	—	M
13	Specific contra-indication(s) or warning(s) such as "irritation", "avoid contact with Eyes", as necessary	—	—	—	M
14	Any hazard warnings (e.g. flammable, etc.)	—	—	—	M
15	The date of the latest revision of the instruction for use	—	—	—	M

"M" indicates mandatory information, "—" indicates non-mandatory information.

Annex A (informative)

Test for fluoride release potential

A.1 Chemicals and solutions

A.1.1 Total ionic strength adjustment buffer (TISAB) solution, in accordance with [6.2.1.1](#).

A.1.2 50 % Diluted TISAB, in accordance with [6.2.1.2](#).

A.1.3 Fluoride standard solutions: $1,1 \times 10^{-6}$, $2,6 \times 10^{-6}$, $5,3 \times 10^{-6}$, $2,6 \times 10^{-5}$, and $5,3 \times 10^{-5}$ mol/l NaF. These are equivalent to 0,02, 0,05, 0,10, 0,50, and 1,01 mg F/l respectively.

NOTE Fluoride standard solution could be commercially available or they could be prepared with sodium fluoride at 0,10 mol/l by appropriate dilution.

A.1.4 Deionized water, in accordance with ISO 3696:1987, grade 2.

A.2 Apparatus

A.2.1 Plastic vial (or beaker or small container), 50 ml or more capacity.

A.2.2 Plastic beaker, 20 ml or more capacity.

A.2.3 Plastic rod (e.g. poly[methyl methacrylate] rod), 150 mm long or longer, with known end cross section area 25 mm^2 to 60 mm^2 (e.g. 6 mm to 8 mm diameter rod). The end surface shall be finished by polishing with 0,045 mm silicon carbide abrasive sheet (e.g. 400 grit SiC paper).

NOTE 1 The rod could have circular cross section or other defined cross-section such as rectangular such that the end surface area could be determined.

NOTE 2 Glass rod could be used instead of poly(methyl methacrylate) rods.

A.2.4 Magnetic stir bars.

A.2.5 Magnetic stir plate.

A.2.6 Plastic test-tube cap, each cap has a hole close to the diameter of the plastic rod ([A.2.3](#)), and capable of holding the plastic rod.

A.2.7 Cardboard, with surface area adequate to cover plastic beaker ([A.2.2](#)) and a hole in each cardboard allows the plastic rod ([A.2.3](#)) to pass through.

A.2.8 Pipette, of nominal volume 5,0 ml and maximum permissible error 0,015 ml and of nominal volume 20,0 ml and maximum permissible error 0,03 ml, in accordance with ISO 648:2008, Class A or AS.

A.2.9 Fluoride ion-selective electrode (F-ISE), in accordance with [6.2.2.6](#).

A.2.10 pH/mV electrometer (pH meter), in accordance with [6.2.2.8](#).

A.3 Procedure

A.3.1 Preparation of calibration curve

Prepare calibration curve in accordance with ISO 19448.

The calibration curve made from the standards shall be sensitive to 0,02 mg F/l and have a slope between -55 mV and -60 mV/ $\log_{10}[F]$ for the standards of 0,05 to 1,00 mg F/l.

A.3.2 Sample Preparation

- a) Pipette 20,0 ml deionized water into a 50 ml beaker.
- b) Add a magnetic stir bar to the beaker.
- c) Place the beaker on the magnetic stir plate.
- d) Mix the fluoride varnish product thoroughly prior to applying the varnish product onto the end of the rod.

NOTE It is important to thoroughly mix the varnish samples prior to painting. It is suggested that the product be dispensed into a small container and thoroughly mixed prior to testing.

- e) Paint the varnish product onto the end of the rod, using the brush supplied with the varnish product if available. Ensure the surface of the rod is completely covered. See [Figure A.1](#) a).

NOTE Always use the same surface area for all samples spread on the surface of the rods.

- f) Suspend the rod in a vertical position with the fluoride varnished covered end of the rod immersed in the beaker containing the deionized water. See [Figure A.2](#).
- g) Start the stirrer and stir for 60 min at $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$. The stir rate shall be 250 r/min to 300 r/min.
- h) Prepare two more samples for a total of three samples.