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Dentistry — Analysis of fluoride concentration in aqueous solutions by use of fluoride ion-selective electrode

Médecine bucco-dentaire — Analyse de la concentration en fluorure dans les solutions aqueuses en utilisant une électrode sélective des ions fluorures

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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).

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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*.

Introduction

Fluoride is used in dentistry primarily for caries prevention. Fluoride is the active ingredient for caries prevention in many dental products such as dentifrices, gels, oral rinses, and fluoride releasing varnishes. The fluoride concentration in these products is an integral part of other standards where the product is intended to be analysed for fluoride content. This document provides methods based on the use of fluoride ion-selective electrode technology for the analysis of the total fluoride content in aqueous samples. The methods describe uses for fluoride ion-selective electrode technology and anticipate that the sample-specific preparations such as digestion, distillation, etc. have been described in the standards specific to product types.

Dentistry — Analysis of fluoride concentration in aqueous solutions by use of fluoride ion-selective electrode

1 Scope

This document specifies test methods for the quantification of fluoride concentrations in dental products including dentifrices, gels, oral rinses, fluoride releasing varnishes, and other fluoride containing products. The methods are based on fluoride ion-selective electrode technology for the analysis of fluoride in aqueous samples derived from dental products.

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

ISO 835, *Laboratory glassware — Graduated pipettes*

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 and ISO 1942 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

4 Principle

This test method covers the determination of fluoride concentration in aqueous solutions derived from dental products. The following test methods are presupposed to use fluoride ion-selective electrodes (F-ISE) .

The sensing element of the fluoride ion-selective electrode consists of a single crystal of europium (II)-doped lanthanum fluoride, LaF_3. When the sensing element is in contact with a solution containing fluoride ions, an electrode potential develops across the sensing element. This potential, which depends on the activity of free fluoride ion in solution $\{F^-\}$, is measured against a constant reference electrode potential with a digital pH/mV meter or ISE (concentration) potentiometer. A potential (E) is developed which is proportional to the logarithm of the activity of the fluoride ion $\{F^-\}$ in solution following the Nernst Equation.

$$E = E_0 + S * \log \{F^-\} \quad (1)$$

where

E is the measured electrode potential;

E_0 is the reference potential (a constant);

{F-} is the fluoride ion activity level in solution;

S is the electrode slope (58,2 mV per decade change of {F-} at 20 °C).

The level of fluoride ion, {F-}, is the activity or "effective concentration" of free fluoride ion in solution. The fluoride ion activity is related to free fluoride ion concentration, [F], by the activity coefficient, γ. The activity coefficient is related to the ionic strength of the solution. For these measurements, the ionic strength of the samples and standards are held constant by the use of total ionic strength adjusting buffer solution (TISAB), which also chelates fluoride-binding cations and establishes the solution pH at the optimal level for the fluoride ion-selective electrode sensitivity. When using TISAB to set the ionic strength of the solution, the electric potential is proportional to the concentration of the fluoride ion in solution. TISAB may come in several variations. Which one to use is specific to the sample preparation procedure. One has to be aware that other interferences may exist and it is up to the user and sample preparation to properly identify them.

A calibration curve is drawn, plotting measured potential versus the log of the fluoride ion concentration of known standard solutions. Then, the potential for the unknown sample is measured and the concentration of F- in the sample can be calculated from the calibration curve.

The sample will have been pretreated as specified for the particular sample type, and the final composition is an aqueous sample of at least 1 ml volume. Samples that contain more than 0,01 mol/l F (190 mg/l F) shall be diluted to concentrations less than 0,01 mol/l F with deionized water.

5 Reagents and materials

5.1 Chemicals to prepare total ionic strength adjusting buffer (TISAB) solution.

5.1.1 Cyclohexylene dinitrilotetraacetic acid (CDTA) or 1,2-diaminocyclohexane-N,N,N',N'-tetraacetic acid.

5.1.2 Acetic acid (CH_3COOH) reagent grade.

5.1.3 Sodium chloride (NaCl) salt.

5.1.4 Sodium hydroxide (NaOH), at 5 mol/l.

5.1.5 Deionized water, in accordance with ISO 3696, grade 2.

5.2 Chemicals to prepare sodium fluoride standards.

5.2.1 Sodium fluoride (NaF), previously dried at least 4 h at 150°C.

5.2.2 Deionized water, in accordance with ISO 3696, grade 2.

6 Apparatus

6.1 Analytical balance, accurate to 0,000 1 g.

6.2 Magnetic stir bar.

6.3 Magnetic stir plate.

6.4 Pipette, of nominal capacity 5,0 ml to smallest scale division: 0,05 ml; 1,0 ml to 0,01 ml; and 0,10 ml to 0,01 ml, in accordance with ISO 835, Class A.

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

6.6 Plastic vial (or beaker or small container), 20 or more ml capacity.

NOTE Fluoride does not react with plastic but does react with glass.

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

7 Preparation

7.1 TISAB solution

7.1.1 In a 1 l beaker with about 500 ml deionized H_2O, dissolve 57 ml CH_3COOH, 58 g NaCl, and 4 g CDTA or 1,2-diaminocyclohexane-N,N,N',N'-tetraacetic acid.

7.1.2 Cool to room temperature and adjust pH to 5,0–5,5 with 5 mol/l NaOH.

7.1.3 Cool to room temperature, transfer to 1 l volumetric flask, and dilute to volume with deionized H_2O.

NOTE Preparation of TISAB solution is described in ASTM D1179–16 [1], 18.1.

TISAB is the buffering system that is used to prepare the sample for analysis via the fluoride ion-selective electrode (F-ISE). The F-ISE has several limitations that can limit its use. It is sensitive only to ionic fluoride in solution, and cannot respond to complexes of fluoride such as calcium-fluoride. Multivalent cations such as calcium, magnesium, iron, aluminium, etc., can form fluoride complexes in solution which reduces the ionic fluoride concentration. Additionally, the F-ISE is sensitive to variations in sample pH and to the ionic strength of the sample. For accurate analyses, the samples and the standards must have the same pH and ionic strength. The fluoride complexes must be disrupted such that all the fluoride in the sample and standard is in its ionic form. To address these limitations, a total ionic strength adjusting buffering system (TISAB) that adjusts the sample pH and ionic strength, and also contains chelators to bind the cations that can form fluoride complexes, is used.

TISAB II® (Orion Research) is available from Thermo Fisher Scientific, Beverly, MA 01915 USA[1)].

7.2 TISAB blank solution

Mix equal volumes of TISAB and deionized water for the blank solution.

7.3 Fluoride standard solutions

7.3.1 1×10^{-2} mol/l NaF standard solution: dissolve 209,9 mg accurately weighed NaF into 200 ml deionized H_2O, then quantitatively transfer to a 500 ml volumetric flask, and dilute to volume with deionized H_2O.

1) This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

