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Dentistry — Polymerization shrinkage: Method for determination of polymerization shrinkage of polymer-based restorative materials

Médecine bucco-dentaire — Rétraction à la polymérisation: Méthode de détermination de la rétraction à la polymérisation des matériaux de restauration à base de polymères

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Foreword

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The committee responsible for this document is ISO/TC 106, *Dentistry*, Subcommittee SC 1, *Filling and restorative materials*.

Introduction

This International Standard specifies a test method for the determination of the polymerization shrinkage of external energy-activated polymer-based restorative materials of Class 2, Group 1 (see ISO 4049) and similar core materials.

Many test methods have been used over many years to determine this property but no International Standard test has so far been adopted. The method specified herein is a simple method that provides reproducible results that will aid users in the comparison of test data. It was developed and verified by a comprehensive interlaboratory test programme comparing it with other methods.

Dentistry — Polymerization shrinkage: Method for determination of polymerization shrinkage of polymer-based restorative materials

1 Scope

This International Standard specifies a test method for the measurement of the polymerization shrinkage of external energy-activated polymer-based restorative materials such as composites and core materials.

The method is not suitable for Class 1 (self-curing, see ISO 4049) polymer-based restorative materials.

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 1183-1, *Plastics — Methods for determining the density of non-cellular plastics — Part 1: Immersion method, liquid pyknometer method and titration method*

ISO 1942, *Dentistry — Vocabulary*

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

ISO 4049, *Dentistry — Polymer-based restorative materials*

ISO 10650 (all parts), *Dentistry — Powered polymerization activators*

3 Terms and definitions

For the purposes of this document, the terms and definitions in ISO 1183-1, ISO 1942, ISO 4049, and the following apply.

3.1
high-viscosity materials
polymer-based restorative materials having little flow so that they hold their shape on moulding

3.2
flowable materials
polymer-based restorative materials having low viscosity so that they do not hold their shape on moulding

4 Test method

4.1 Principle

The polymerization shrinkage of external energy-activated polymer-based restorative materials is determined using density determinations in accordance with the buoyancy method (Archimedes' principle). This test method accords with method A (immersion method), described in general terms in ISO 1183-1.

Ensure that test conditions such as temperature, duration of exposure, and distance between light guide and the test specimen are controlled and reproducible. Pre-test storage conditions of the polymer prior to its measurement are also specified to ensure the maximum achievable polymerization in the test

conditions so that the most complete possible shrinkage of the materials will be achieved. This way, differences in the rate of polymerization will, to a large extent, be balanced out.

4.2 General

Perform all measurements at a room temperature of (23 ± 2) °C.

Store all specimens at a temperature of (23 ± 2) °C for at least 30 min before the start of measurement.

Perform all measurements of the unpolymerized specimens under yellow light.

NOTE 1 Yellow light may be created by filtering ambient lighting with a suitable filter[1)].

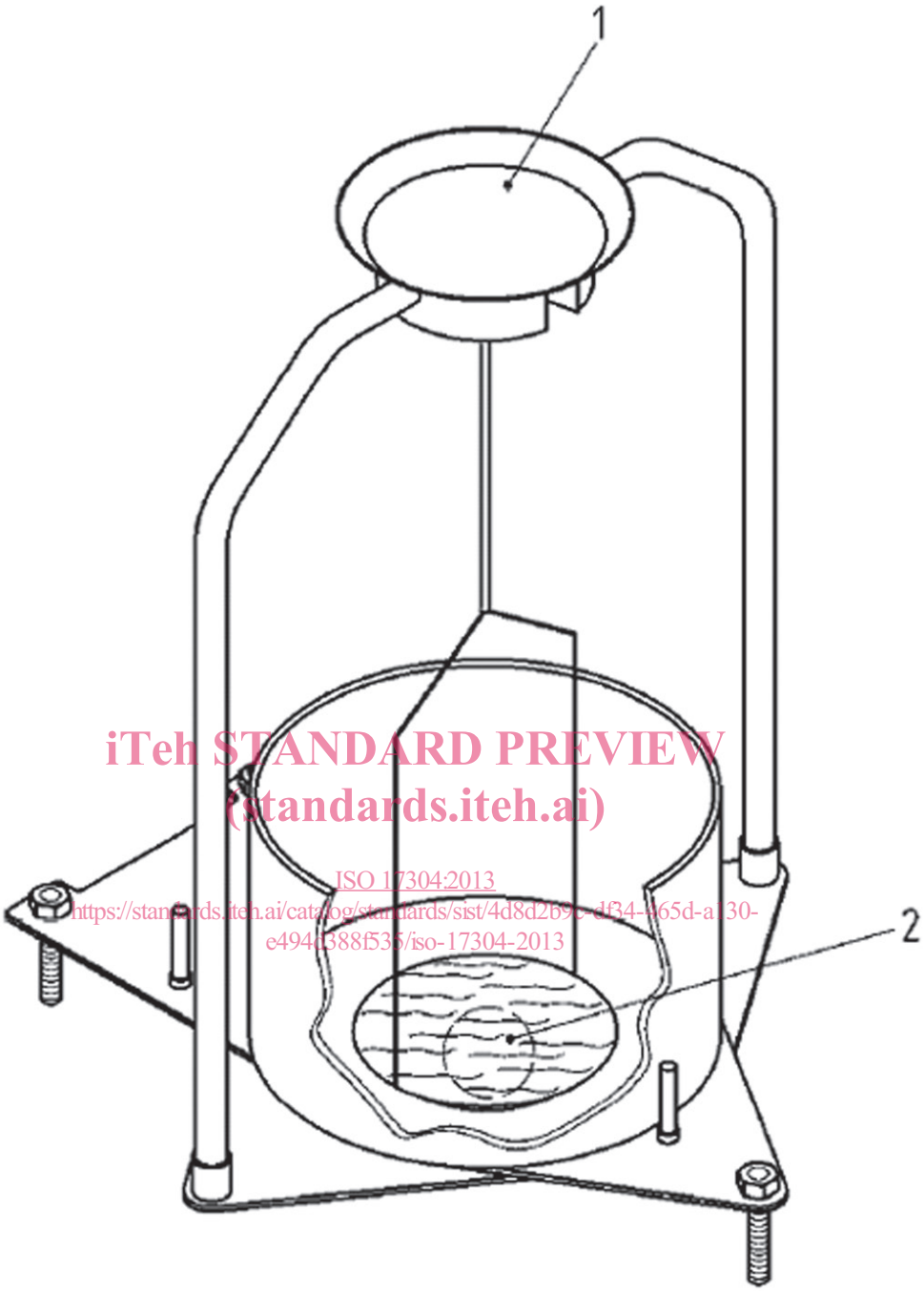
When handling test materials, wear appropriate rubber gloves, e.g. latex or nitrile.

The temperature fluctuation during the measurement of the unpolymerized specimens and the polymerized specimens shall be less than 1,0 °C.

NOTE 2 Measurements can be performed at temperatures between 21 °C and 25 °C. Temperature variations during measurement below 1,0 °C do not significantly influence the results.

Perform the corresponding density measurements for the unpolymerized and the polymerized specimens on the same day.

1) Details of suitable products are available from the secretariat of ISO/TC 106.



Key

1 position 1: for the specimen in air (dry)

2 position 2: for the specimen in the buoyancy medium

Figure 1 — Example of a density determination apparatus

4.3 Materials and reagents

4.3.1 General reagent — water, use water prepared in accordance with ISO 3696 Grade 3.

4.3.2 Sodium lauryl sulphate, with at least 99,0 % (mass concentration) purity.

NOTE The CAS number of sodium lauryl sulphate is 151–21–3.

4.3.3 Test material — External-energy activated polymer-based restorative material or polymeric core material.

4.4 Apparatus

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

4.4.2 Density determination apparatus, consisting essentially of a platform, beaker, pan hanger assembly, and thermometer (see Figure 1).

NOTE This equipment may be obtained from scale manufacturers as a special accessory (density determination kit).

4.4.3 Thermometer, accurate in the range 20 °C to 30 °C, graduated in steps of 0,1 °C.

4.4.4 External energy source, with a radiant exitance of at least 500 mW/cm^2 in the range of 400 nm to 515 nm [determined in accordance with ISO 10650 (all parts)] and recommended by the manufacturer of the test material for use with that material.

4.4.5 Vacuum pump, with pressure gauge (manometer), accurate to 10 mbar.

4.4.6 Desiccator, with a valve and capable of being connected to the vacuum pump (4.4.5)

4.4.7 Oven, capable of being maintained at (37 ± 2) °C.

4.4.8 Spatula.

4.4.9 Transfer medium, suitable for the unpolymerized paste, e.g. microscope slide.

4.4.10 Volumetric flask, of size 500 ml, with stopper.

4.4.11 Smooth, non-absorbent, stable base, e.g. mixing (conditioner) block.

4.4.12 Glass dish, for use with flowable materials, fitting to the measurement pans of the determination apparatus (4.4.2), e.g. diameter of about 20 mm and a height of about 10 mm.

4.5 Buoyancy medium

4.5.1 Determination of immersion depth

The immersion depth of the lower weighing pan of the density determination apparatus (4.4.2) shall be at least 20 mm to ensure complete immersion. Immersion depth is measured from the upper edge of the weighing pan to the meniscus of the buoyancy medium.

For flowable materials, the specimen and the dish shall be completely immersed.

NOTE The density determination apparatus supplied by various manufacturers differ with regard to the height and diameter of their water bowls. It is necessary to establish a standard immersion depth for the lower weighing pan in the buoyancy medium to ensure complete immersion.

4.5.2 Preparation of the buoyancy medium

Weigh (5,000 ± 0,001) g of sodium lauryl sulphate (4.3.2), place it into the volumetric flask (4.4.10), and then add water (4.3.1) until a final weight of (500 ± 0,001) g is reached. Seal the flask and shake it until

a homogeneous solution is achieved, as determined by visual inspection without magnification. Prepare the buoyancy medium at least 24 h prior to testing.

NOTE 1 This solution can be stored for four weeks at room temperature.

On the day of the test, pour the necessary amount of buoyancy medium into the water bowl of the determination apparatus in accordance with the distance specifications in 4.5.1 and place it in the desiccator (4.4.6).

Degas the buoyancy medium by evacuating the desiccator at a temperature of (23 ± 2) °C using the vacuum pump (4.4.5). Immediately after a vacuum pressure of (50 ± 10) mbar is attained, close the desiccator vacuum port. After (20 ± 5) min, carefully ventilate the dessicator.

NOTE 2 This pressure was chosen so that the water does not boil.

Afterwards, the buoyancy medium shall be kept at a temperature of (23 ± 2) °C for at least 2 h.

Read the density of the buoyancy medium for the current measuring temperature from Table 1.

NOTE 3 The density of water is applied instead of the precise density of the buoyancy medium. The dependence on temperature will thereby be taken into account. The accuracy of the measured polymerization shrinkage will thereby not be impaired because the differences in the respective densities balance each other out.

4.5.3 Number of specimens

For the testing of unpolymerized pastes, prepare 6 specimens of 1,0 g each.

For the testing of polymerized pastes, prepare 12 specimens of 0,5 g each.

4.6 Preparatory treatment of the test material

4.6.1 General

The dispensing, movement, and storage of the unpolymerized material shall take place under yellow light. See 4.2.

The first 3 mm of the expressed material shall not be used.

4.6.2 Test material pastes in syringes

Deploy test material pastes in syringes directly without further preparatory treatment prior to the determination of the polymerization shrinkage.

4.6.3 Test material pastes in single-dosage containers

Pre-treat test material pastes in single-dosage containers (e.g. compules, carpules) before use in the following way.

Squeeze the material out slowly and without bubbles onto the smooth, non-absorbent, stable base (4.4.11). Store this block and material for two days at (37 ± 2) °C in the oven (4.4.7) with light excluded.

Condition the material at a temperature of (23 ± 2) °C for 1 h before measurement.

NOTE This period of storage serves to remove any entrapped air bubbles from the material that may have been introduced during the expression or movement of the material.