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Dentistry — Dental furnace —

Part 3: Test method for the evaluation of high temperature sintering furnace measurement with a separate thermocouple

Médecine bucco-dentaire — Fours dentaires —

Partie 3: Méthode d'essai pour l'évaluation du mesurage des hautes températures de frittage au moyen d'un thermocouple externe

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Foreword

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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 document 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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This document was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 2, *Prosthodontic materials,* 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).

A list of all parts in the ISO 13078 series can be found on the ISO website.

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

In dentistry, sintering furnaces are used for sintering restorations made from oxide ceramics and from sintered metal. Significantly higher temperatures than those for firing dental ceramic masses containing silicates are necessary, for example, zirconium oxide (ZrO_2) is typically sintered at a temperature of up to 1 700 °C.

The sintering temperature is of vital importance for the properties of the sintered material. Incorrect sintering temperatures can result in low strength, discrepant colouration or low ageing resistance. Furthermore, a poor accuracy of fit owing to excessively low or uneven shrinkage can occur. Too high a sintering temperature generally results in a larger grain size and can lead to a softening and consequently a deformation of the restoration. Too low a sintering temperature results in an inadequate sintering quality and possibly residual porosity.

Dentistry — Dental furnace —

Part 3: Test method for the evaluation of high temperature sintering furnace measurement with a separate thermocouple

1 Scope

This document specifies a test method for the calibration of resistance-heated high temperature sintering furnaces that are suitable for the sintering of dental restorations in the temperature range up to 1 700 °C.

NOTE A test method for the calibration of dental furnaces that are suitable for the heat treatment of silica-based dental ceramic restorations in the temperature range between 600 °C and 1 050 °C is specified in ISO 13078:2013. ISO 13078:2013 does not include the calibration of sintering furnace used for sintering of oxide ceramics or sintered metal, in whose firing chamber restorations are sintered at temperatures of 1 000 °C to 1 700 °C.

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 1942, *Dentistry — Vocabulary*

ISO 6872, *Dentistry — Ceramic materials*

IEC 60584-1:2013, *Thermocouples — Part 1: EMF specifications and tolerances*

IEC 60584-3, *Thermocouples — Part 3: Extension and compensating cables — Tolerances and identification system*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1942, ISO 6872 and IEC 60584-1:2013 and the following apply.

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

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

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

3.1
heating rate
rate of temperature increase

Note 1 to entry: The heating rate shall be expressed in degrees Celsius per minute (°C/min).

3.2
holding time
time span in which the desired temperature of the *sintering* (3.5) furnace is retained up to the time specified by the manufacturer, beginning from the time point at which the display on the sintering furnace indicates that the sintering furnace has reached the desired temperature

3.3
high temperature sintering furnace
sintering (3.5) furnace, in whose firing chamber restorations are sintered at temperatures of 1 000 °C to 1 700 °C

3.4
oxide ceramic
ceramic produced from a crystalline feedstock powder at high temperatures via a *sintering* (3.5) process

Note 1 to entry: All ceramics which do not contain SiO_2 and which are exclusively oxidic are referred to as oxide ceramics.

Note 2 to entry: In contrast to this, ceramics containing SiO_2 are referred to as silicate ceramics. If ceramics contain not only oxygen as an electronegative component but also, for example, carbon or nitrogen, they shall be referred to as non-oxide ceramics. Only the oxide ceramics zirconium oxide and aluminium oxide as well as composites of these are common in dental technology at present.

3.5
sintering
permanent consolidation of a moulding body from a compacted powder aggregate material by means of a firing process, in which a decrease in the porosity, an increase in the density and a (sinter) shrinkage occur

Note 1 to entry: The temperature here shall be sufficiently high to cause densification but shall not be so high that a deformation of the moulding body occurs.

3.6
sintered metal
blanks produced from metal powder for milling via CAD/CAM technology for restorations made from base metal alloys, which are sintered in inert gas (argon) at temperatures of 1 200 °C to 1 400 °C

4 Measurement and test method

4.1 General

This document describes the calibration of resistance-heated high temperature sintering furnaces by means of a separate thermocouple. The furnaces shall be calibrated ex works in the same way for all manufacturers at a temperature of 1 500 °C. In addition, the deviation in the actual temperature at 1 000 °C shall be determined and a maximum permissible deviation specified.

4.2 Test devices and test equipment

4.2.1 Thermocouple fixture

The thermocouple fixture shall be made from a lightweight material resistant to high temperatures that is white in colour. The thermocouple shall be set up corresponding to Figure 1.

a) material: polycrystalline ceramic fibres, for example, Al_2O_3: 75 % to 85 %; SiO_2: 15 % to 25 %; about 400 kg/m^3;

b) height: X = (20 ± 2) mm (depending on the furnace chamber);

c) diameter: (40 ± 2) mm (depending on the furnace chamber).

4.2.2 Thermocouple

The thermocouple has the following specifications:

a) thermocouple Pt10Rh-Pt (type S) according to IEC 60584-1:2013, with a limit deviation in the thermoelectric voltage of class 1 according to IEC 60584-1:2013, Table 12;

NOTE Pt13Rh-Pt (type R) according to IEC 60584-1:2013 is permissible as an alternative.

b) diameter of the legs: 0,5 mm according to IEC 60584-3;

c) diameter of the head: (1,5 ± 0,5) mm;

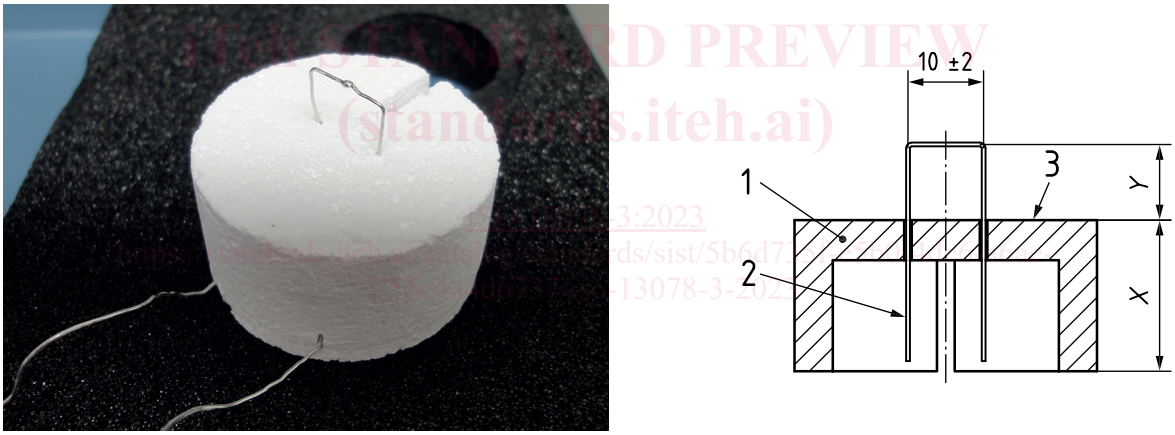
d) distance of the legs: (10 ± 2) mm;

e) height above the thermocouple fixture (depending on the furnace chamber, at the location of the object to be fired), for example, (10 ± 2) mm.

The thermocouple shall be located centrally at a height Y = (10 ± 2) mm above the object level (see Figure 1). The horizontal distance of the connecting wires protruding out of the thermocouple fixture is (10 ± 2) mm.

Alternatively, the thermocouple can be introduced directly into the base.

Dimensions in millimetres



Key

X height of the thermocouple fixture
Y height of the thermocouple above the object level
1 thermocouple fixture
2 thermocouple
3 object level

Figure 1 — Layout of the thermocouple on the thermocouple fixture

4.2.3 Compensating cable

The compensating cable for the thermocouple shall have a limiting deviation of the compensating cable of class 2 in accordance with IEC 60584-3. The connection point shall be compensated by the measuring device.

4.2.4 Temperature indicating device

The temperature indicating device shall be calibrated to ±1,2 °C between 400 °C and 1 550 °C (e.g. HP Data Logger or Ezecal[1)]).

4.2.5 High temperature sintering furnace

4.3 Implementation

4.3.1 Layout of the thermocouple

The thermocouple shall be installed centrally in the firing chamber of the high temperature sintering furnace or in the position where the firing material is located in accordance with the manufacturer's specifications.

4.3.2 Heating phase

Before beginning the calibration, it shall be established that the high temperature sintering furnace is ready for operation.

The heating rate during the heating phase shall be minimum 10 °C/min. Calibration shall take place under ambient conditions (no negative pressure or vacuum).

4.3.3 Test temperature 1

The high temperature sintering furnace shall be brought to a first test temperature of 1 000 °C.

4.3.4 Holding time 1

The holding time begins when the display on the high temperature sintering furnace indicates that the set test temperature has been reached. The holding time is 10 min.

4.3.5 First measurement

During the first holding time, the temperature values determined via the external thermocouple shall be read off and noted after 0 s, 15 s, 60 s, 120 s and 600 s.

4.3.6 Test temperature 2

The high temperature sintering furnace shall be brought to a second temperature of 1 500 °C.

4.3.7 Holding time 2

The holding time begins when the display on the high-temperature sintering furnace indicates that the set test temperature has been reached. The holding time is 10 min.

4.3.8 Second measurement

During the second holding time, the temperature values determined via the external thermocouple shall be read off and noted after 0 s, 15 s, 60 s, 120 s and 600 s.

1) HP Data Logger and Ezecal are examples of suitable products available commercially. This information is given for the convenience of the users of this document and does not constitute an endorsement of this product by ISO.