INTERNATIONAL STANDARD

ISO 20274

First edition
2017-07

Vitreous and porcelain enamels — Preparation of samples and determination of thermal expansion coefficient

Emaux vitrifiés — Préparation d'échantillons d'émail et détermination du coefficient de dilatation thermique

Reference number
ISO 20274:2017(E)

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

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 107, *Metallic and other inorganic coatings*.

Introduction

The thermal expansion coefficient of enamel and the relevant substrate is an important material characteristic, as it provides information on the stress ratios in the composite material.

Vitreous and porcelain enamels — Preparation of samples and determination of thermal expansion coefficient

1 Scope

This document specifies the procedures for the preparation of enamel samples for measurement of the thermal length change and calculation of the thermal expansion coefficient.

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 7991, *Glass — Determination of coefficient of mean linear thermal expansion*

ISO 19496-1, *Vitreous and porcelain enamels — Terminology — Part 1: Terms and definitions*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 19496-1 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 http://www.iso.org/obp

4 Apparatus

4.1 Standard laboratory instruments, instruments in accordance with ISO 7991 and 4.2 to 4.4.

NOTE An optical dilatometer can be used as an alternative to a pushrod dilatometer.

4.2 Casting mould made from ceramic, for example, boat made from corundum, porcelain, or casting mould made from metal, for example, tool steel (see Annex A).

4.3 Melting crucible made from non-contaminating material, for example, corundum, porcelain, with a height of about 55 mm and a diameter of approximately 40 mm.

4.4 Laboratory furnace, in the temperature range from 800 °C to 1 100 °C controllable to ±5 °C.

5 Requirements for the test specimen

The test specimen shall be rod-shaped and straight with either

— an approximately circular cross section with a diameter constant to ±1 mm over its length, or

— an approximately square cross section with an edge length constant to ±1 mm over its length.

The diameter or edge length of the test specimen shall correspond ±1 mm to the diameter or edge length of the reference sample.

The faces shall be plane-parallel and vertical to the longitudinal axis of the specimen to maximum ±3°. The initial length shall be ±0,5 mm equal to the initial length of the reference sample.

The dimensions shall correspond to the requirements of the dilatometer used and the reference sample.

6 Preparation of the test specimens

6.1 General

The test specimen shall be sufficiently stress-free by tempering so that no dip occurs in the measuring diagram when measuring the expansion coefficient.

NOTE An absence of stress can, for example, be attained by heating the test specimen to approximately 30 °C above the transformation temperature, followed by slow cooling in the furnace at approximately 2 °C/min to approximately 150 °C below the transformation temperature and then to the room temperature in draught-free air.

The production process for a test specimen prepared according to this document shall be denoted as follows:

G production by casting

Z production by drawing

S production by sintering

6.2 Production by casting

The enamel frit shall be melted in the melting crucible (4.3) in the laboratory furnace (4.4). To prevent evaporation losses, melting temperatures and holding times that are appropriate to allow for pouring out into the mould should be used; excessive melting temperatures and holding times will distort results. The melting stock shall be filled into the casting mould (4.2) while avoiding air inclusions.

If using a casting mould made from ceramic, this shall be coated beforehand with a release agent, for example, kaolin, and preheated to approximately 500 °C. The test specimen in the casting mould shall then be cooled down sufficiently slowly so that it solidifies without cracks (see also 6.1). The test specimen shall be free of release agent and cut and polished to the required dimensions, ensuring the ends are parallel to each other.

If using a casting mould made from metal, the specimen in the mould shall be cooled in air until solidification. A suitable ceramic plate shall be placed in the mould and lifted and supported with the mould so that the specimen remains on the ceramic plate. The plate with the specimen shall then be placed immediately in the laboratory furnace (4.4), heated to approximately 30 °C above the transformation temperature and cooled down crack-free (see also 6.1). The test specimen shall be cut to the required length and ground plane-parallel.

6.3 Production by drawing out of the melt

The frit shall be melted in the crucible (4.3) in the laboratory furnace (4.4) at a temperature between 900 °C and 980 °C adapted to the hardness of the enamel.

NOTE 1 Too high a heating is disadvantageous as it seriously impedes the subsequent drawing of the rod for the test specimen production. In general, the temperature is reached if a smooth enamel surface has resulted in the melting crucible.

After the frit has reached the firing temperature, additional frit is added to the melting crucible and melted a second time, as the drawing is difficult if the amount of liquid enamel is inadequate. The melting crucible may not be filled more than three times maximum. After preparing the melt, the melting crucible (4.3) shall be removed from the laboratory furnace (4.4) within 15 s. The melting crucible shall be held with crucible tongs and now a glass or metal rod is immersed slightly in to the

liquid enamel and is rotated until the glass fuses around the rod d [see Figure B.1 a) and Figure B.1 b)]. In this way, air inclusions can be prevented. The direction of turning may not be changed. It is necessary to wait until a small melt ball has resulted, this indicating the end of the fusion [see Figure B.1 c)]. The rod shall then be drawn up gently, a pillar-shaped test specimen resulting through solidification of the melt [see Figure B.2 a) to Figure B.2 c)].

NOTE 2 The thickness of the test specimen depends on the drawing speed.

The rod shall be drawn from the centre in order to extensively avoid a contamination of the test specimens by the vessel material.

The rod with the drawn test specimen shall be removed from the melt after reaching the required length.

The test specimen drawn in this way shall not flatten and shall exhibit a uniform diameter (see Clause 5). After cooling, the test specimen shall be cut and polished to the required dimensions ensuring the ends are parallel to each other, and relieved by tempering (see 6.1).

NOTE 3 The risk of flattening the rod drawn from the melt is greater the further the drawn-out mass becomes from the centre of the crucible in the direction of the crucible wall.

NOTE 4 To remove the rod with the resultant test specimen from the melt, the rod can be held at the bottom with crucible tongs, transferred and broken off [see Figure B.3 a) and Figure B.3 b)].

6.4 Production by sintering

To produce a mould, an impression (negative) of a test specimen (positive) shall be made in a boat filled with, for example, kaolin. The mould obtained in this way shall be filled with the sintering enamel, for example, thick slip or application-ready powder. After drying, sintering shall be carried out for approximately 15 min in the laboratory furnace (4.4) at the firing temperature within a tolerance of ±15 °C. The resulting test specimen shall be left to cool, free of release agent, cut and polished to the required dimensions, ensuring the ends are parallel to each other, and relieved by tempering (see 6.1).

7 Determination of the thermal expansion coefficient

Before beginning the measurement of the length change, the initial length of the test specimen shall be determined at 0,1 mm.

The determination of the thermal expansion coefficient shall be conducted according to ISO 7991.

The measuring results shall be corrected owing to the differences in the expansion response between the specimen and specimen holder or reference sample as well as on account of the inhomogeneous temperature field.

NOTE 1 The single-point method applied according to ISO 7991 is a conventional method. Nowadays, it is normal to correct each measuring point gained by thermal expansion in comparison to the calibration material using computers.

Depending on the application temperature range and expansion coefficient, one of the materials listed in Table 1 shall be used as calibration material, whereby the thermal expansion coefficient of the calibration material may not deviate from the measured test specimen by more than 25 %.

An inert atmosphere shall be used for measurement when using copper.

NOTE 2 Gold is suitable as a low temperature calibration material (–183 °C to 16 °C: 396×10^{-7} K^{-1}, 16 °C to 100 °C: 429×10^{-7} K^{-1}).

The heating rate shall be ≤ 10 °C/min and selected so that no temperature gradient is present in the test specimen. A heating rate of 5 °C/min is recommended.

The measurement may be started between 18 °C and 28 °C for practical considerations. A start temperature of 20 °C should be selected.