INTERNATIONAL STANDARD

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Vitreous and porcelain enamels — Determination of resistance to chemical corrosion —

Part 3: Determination of resistance to chemical corrosion by alkaline liquids using a hexagonal vessel or a tetragonal glass bottle

Émaux vitrifiés — Détermination de la résistance à la corrosion chimique —

Partie 3: Détermination de la résistance à la corrosion chimique par des liquides alcalins dans un récipient hexagonal ou une bouteille en verre tétragonale

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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

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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 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).

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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 107, *Metallic and other inorganic coatings*.

This second edition cancels and replaces the first edition (ISO 28706-3:2008), which has been technically revised.

A list of all parts in the ISO 28706 series can be found on the ISO website.

Introduction

Corrosion of vitreous and porcelain enamels by aqueous solutions is a dissolution process. The main component of the enamel, SiO_2, forms a three-dimensional silica network. After hydrolysis, it decomposes and forms silicic acid or silicates. These are released into the attacking medium. Other components, mainly metal oxides, are hydrolyzed as well and form the corresponding hydrated metal ions or hydroxides. All corrosion products are more or less soluble in the attacking medium. The whole process results in a loss in mass per unit area.

For some aqueous solutions, the attack on the enamel proceeds linearly during the corrosion time; for other aqueous solutions, the attack on the enamel proceeds in a logarithmic manner during the corrosion time. Only for the first series of solutions can a scientifically exact rate of loss in mass per unit area ($g/m^2 \cdot h$) be calculated as well as a corrosion rate (mm/year).

The most important parameters influencing aqueous corrosion of the enamel are the enamel quality, the temperature and the pH-value. Inhibition effects resulting from the limited solubility of silica can also contribute. The following list describes different types of enamel attack for different corrosion conditions.

a) In aqueous alkali solutions like 0,1 mol/l NaOH (see ISO 28706-4:2016, Clause 9), the silica network of the enamel is considerably attacked at 80 °C. Silicates and most of the other hydrolyzed components are soluble in the alkali. Attack proceeds linearly during regular test times. Therefore, test results are expressed in terms of a rate of loss in mass per unit area (mass loss per unit area and time) and a corrosion rate (millimetres per year).

b) At room temperature, in weak aqueous acids like citric acid (see ISO 28706-1:2008, Clause 9) or also in stronger acids like sulfuric acid (see ISO 28706-1:2008, Clause 10), there is only minor attack on the silica network of the enamel. Other constituents are leached to some extent from the surface. Highly resistant enamels will show no visual change after exposure. On less resistant enamels, some staining or surface roughening will occur.

c) In boiling aqueous acids (see ISO 28706-2), the silica network of the enamel is being attacked, and silica as well as the other enamel components are released into solution. However, the solubility of silica in acids is low. Soon, the attacking solutions will become saturated with dissolved silica and will then only leach the surface. The acid attack is inhibited and the rate of corrosion drops markedly.

NOTE The glass test equipment also releases silica by acid attack and contributes to the inhibition of the corrosion.

Inhibition is effectively prevented in vapour phase tests. The condensate formed on the test specimen is free of any dissolved enamel constituents.

Examples of enamel corrosion proceeding in a logarithmic manner [see 1)] and linearly [see 2)] are:

1) Boiling citric acid (see ISO 28706-2:2017, Clause 11) and boiling 30 % sulfuric acid (see ISO 28706-2:2017, Clause 12)

Since only minute amounts of these acids are found in their vapours, the test is restricted to the liquid phase. The attack is influenced by inhibition effects, and corrosion depends on the time of exposure. Therefore, test results are expressed in terms of loss in mass per unit area; no rate of loss in mass per unit area is calculated.

2) Boiling 20 % hydrochloric acid (see ISO 28706-2:2017, Clause 13)

Since this is an azeotropic boiling acid, its concentration in the liquid and the vapour phase are identical, and liquid phase testing need not be performed. Vigorous boiling supplies an uninhibited condensate, and the attack proceeds linearly with time of exposure. Therefore, test results are only expressed in terms of rate of loss in mass per unit area (mass loss per unit area and time) and the corrosion rate (millimetres per year).

d) At high temperatures, with tests in the liquid phase under autoclave conditions (see ISO 28706-5), aqueous acid attack is severe. To avoid inhibition, the test time is restricted to 24 h and the ratio of attacking acid to attacked enamel surface is chosen so that it is comparatively high (similar to that in a chemical reaction vessel). In addition, only low-silica water is used for the preparation of test solutions. Under these conditions, attack will proceed linearly with time of exposure. Therefore, test results with 20 % hydrochloric acid (see ISO 28706-5:2010, Clause 8), artificial test solutions (see ISO 28706-5:2010, Clause 10) or process fluids (see ISO 28706-5:2010, Clause 11) are also expressed in terms of a rate of loss in mass per unit area (loss in mass per unit area and time).

e) In boiling water (see ISO 28706-2:2017, Clause 14), the silica network is fairly stable. The enamel surface is leached and silica is dissolved only to a small extent. This type of attack is clearly represented by the vapour phase attack. In the liquid phase, some inhibition can be observed with highly resistant enamels. However, if the enamel being tested is weak, leached alkali from the enamel can raise pH-values to alkaline levels, thus increasing the attack by the liquid phase. Both liquid and vapour phase testing can give valuable information.

f) Since the attack may or may not be linear, the results are expressed only in terms of loss in mass per unit area, and the test time should be indicated.

g) For standard detergent solution (see Clause 10), it will not be certain whether the linear part of the corrosion curve will be reached during testing for 24 h or 168 h. Calculation of the corrosion rate is therefore not included in the test report.

h) For other acids (see ISO 28706-2:2017, Clause 16) and other alkaline solutions (see Clause 11 and ISO 28706-4:2016, Clause 11), it will also not be known if a linear corrosion rate will be reached during the test period. Calculation of the corrosion rate is therefore not included in the test reports of those parts of the ISO 28706 series.

For vitreous enamels fired at temperatures below 700 °C, the test parameters (media, temperatures and times) of this document are not appropriate. For such enamels, for example, aluminium enamels, other media, temperatures and/or times should be used. This can be done following the procedures described in the clauses for “other test solutions” in ISO 28706-1, ISO 28706-2, this document (ISO 28706-3) and ISO 28706-4.

Vitreous and porcelain enamels — Determination of resistance to chemical corrosion —

Part 3:
Determination of resistance to chemical corrosion by alkaline liquids using a hexagonal vessel or a tetragonal glass bottle

1 Scope

This document describes a test method for the determination of the resistance of vitreous and porcelain enamelled articles to attack by alkaline liquids at temperatures between 25 °C and 95 °C. The apparatus used is a hexagonal vessel in which six enamelled specimens or a tetragonal glass bottle in which four enamelled specimens are simultaneously tested.

NOTE 1 The resistance to any alkaline liquid can be determined. However, the test method was originally used for the determination of the resistance to hot detergent solutions, within the neutral and alkaline range, used for washing textiles.

NOTE 2 Since detergents are continually subject to alterations in their composition, a standard test solution is specified which, in respect to its alkalinity, wetting properties and complexing behaviour, can be considered as a typical composition for the detergents present on the market. The pH value and alkalinity of the standard test solution depend on the proportions of sodium tripolyphosphate, sodium carbonate and sodium perborate present; sodium tripolyphosphate acts simultaneously as a complexing agent. The wetting properties of the standard test solution are obtained by the addition of alkylsulfonate. A higher sodium perborate content is not considered necessary since the effect of oxygen on enamel is unimportant and an increase in the perborate content does not cause any significant alteration in the alkalinity of the standard test solution. The testing of different enamels using this standard test solution and other test solutions (including 5 % sodium pyrophosphate solution) has justified the use of this standard test solution for determining the resistance of enamels to hot detergent solutions.

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 48, *Rubber, vulcanized or thermoplastic — Determination of hardness (hardness between 10 IRHD and 100 IRHD)*

ISO 3585, *Borosilicate glass 3.3 — Properties*

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

ISO 4799, *Laboratory glassware — Condensers*

ISO 28764, *Vitreous and porcelain enamels — Production of specimens for testing enamels on sheet steel, sheet aluminium and cast iron*

3 Terms and definitions

No terms and definitions are listed in this document.

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 Principle

Six (6.1) or four (6.2) similarly enamelled specimens are simultaneously exposed to attack by an alkaline liquid under specified conditions of temperature and time, the solution being continuously stirred during the test.

The loss in mass is determined and used to calculate the rate of loss in mass per unit area.

NOTE In order to correspond to the conditions of a washing machine used in practice, the alkaline liquid is stirred during the test. The solution is cold when put into the vessel and is heated to the desired temperature in the vessel.

5 Reagents

During the determination, use only reagents of recognized analytical grade, unless otherwise specified.

5.1 Water, conforming to the requirements of grade 3 of ISO 3696, i.e. distilled water or water of equivalent purity.

5.2 Degreasing solvent, such as ethanol, or water (5.1) containing a few drops of liquid detergent, suitable for cleaning the test apparatus and test specimens.

5.3 Sodium tripolyphosphate ($Na_5P_3O_{10}$).

5.4 Sodium carbonate (Na_2CO_3), anhydrous.

5.5 Sodium perborate, hydrated ($NaBO_2 \cdot H_2O_2 \cdot 3H_2O$).

5.6 Sodium silicate, containing about 81 % (by mass) of Na_2SiO_3.

5.7 Alkylsulfonate [$CH_3(CH_2)_x - C(SO_2Na)H - (CH_2)_3 - CH_3$].

5.8 Acetic acid solution, volume concentration 50 ml/l, for cleaning the test apparatus and test specimens.

6 Apparatus and materials

6.1 Hexagonal vessel apparatus.

6.1.1 The apparatus (see Figures 1 to 4) consists of a hexagonal vessel having a circular opening on each side. A specimen is pressed against each of these openings by means of gripping plates which are held in place by wing nuts, sealing rings being placed between the vessel and the specimens. A lid having four holes, for a paddle stirrer, two immersion heaters and a temperature-controlling device, is screwed on to the vessel, a sealing ring being placed between the vessel and the lid. The paddle stirrer, immersion heaters and temperature-controlling device are fixed such that their distance from the bottom of the vessel is 30 mm.

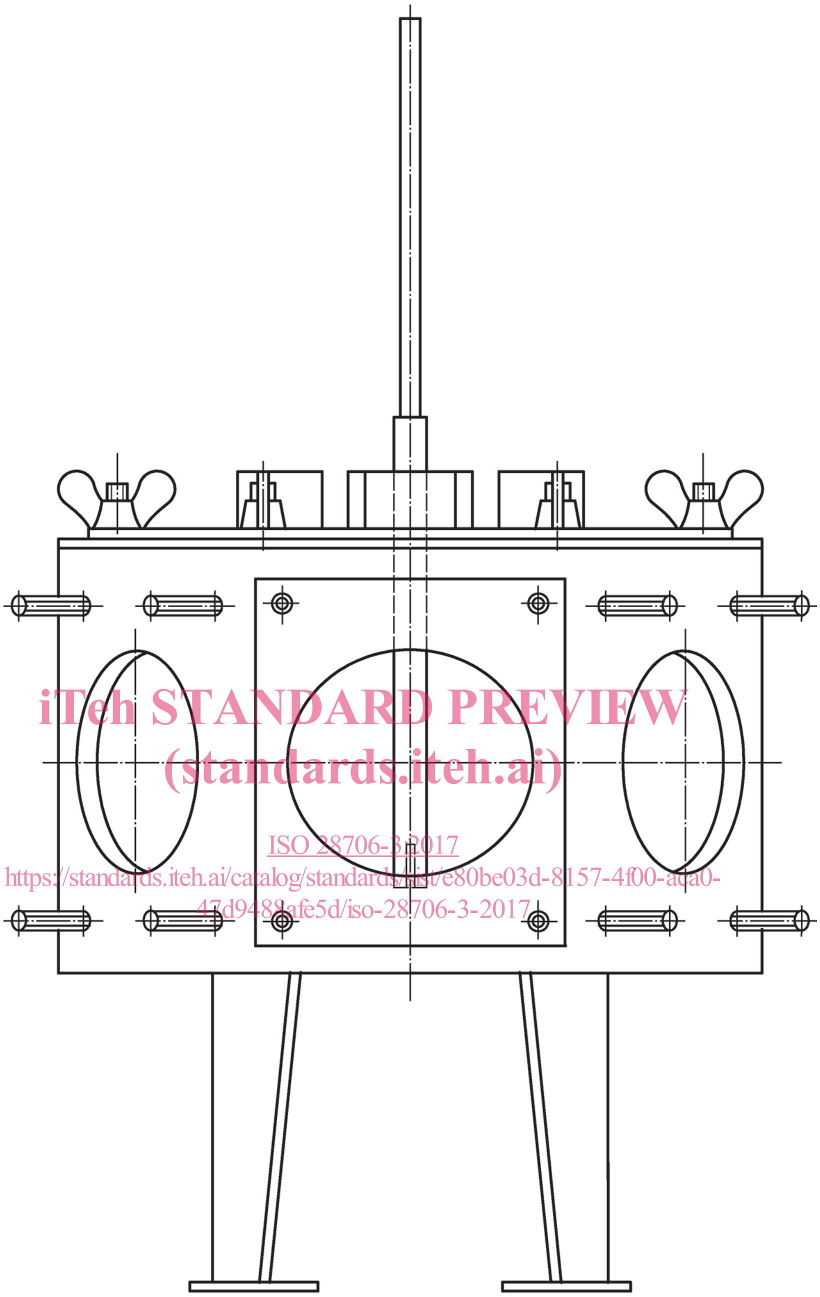
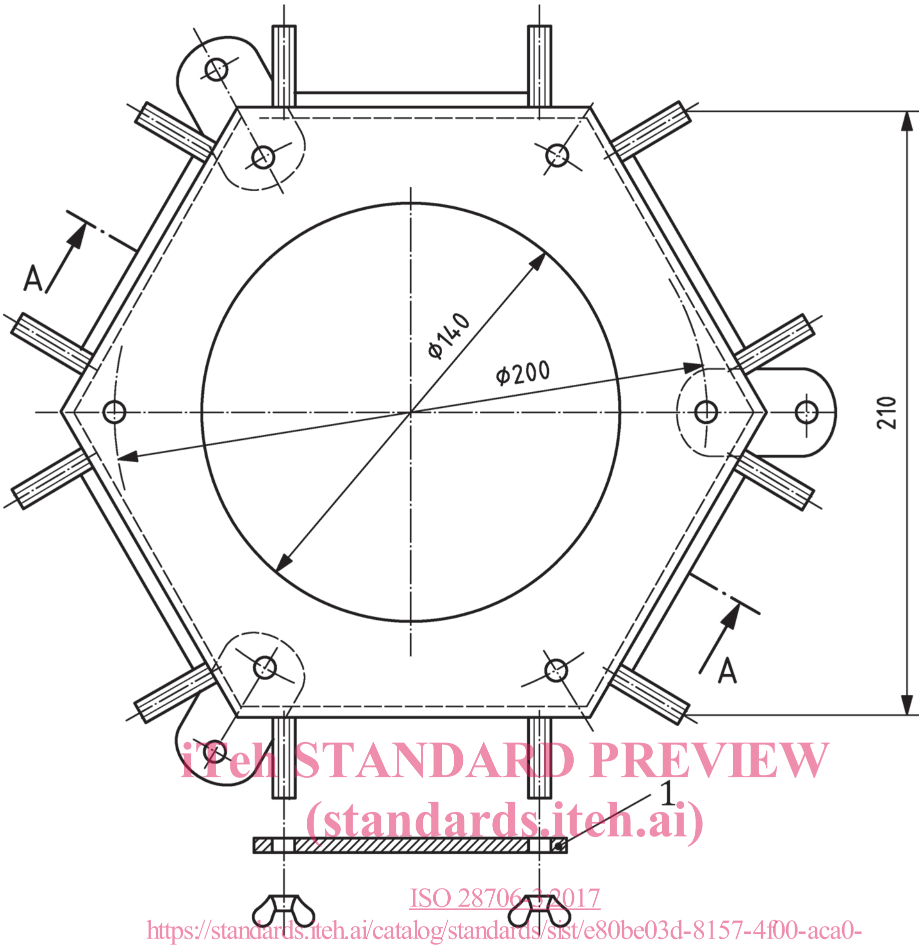


Figure 1 — Hexagonal vessel with lid, stirrer and gripping plate

Dimensions in millimetres

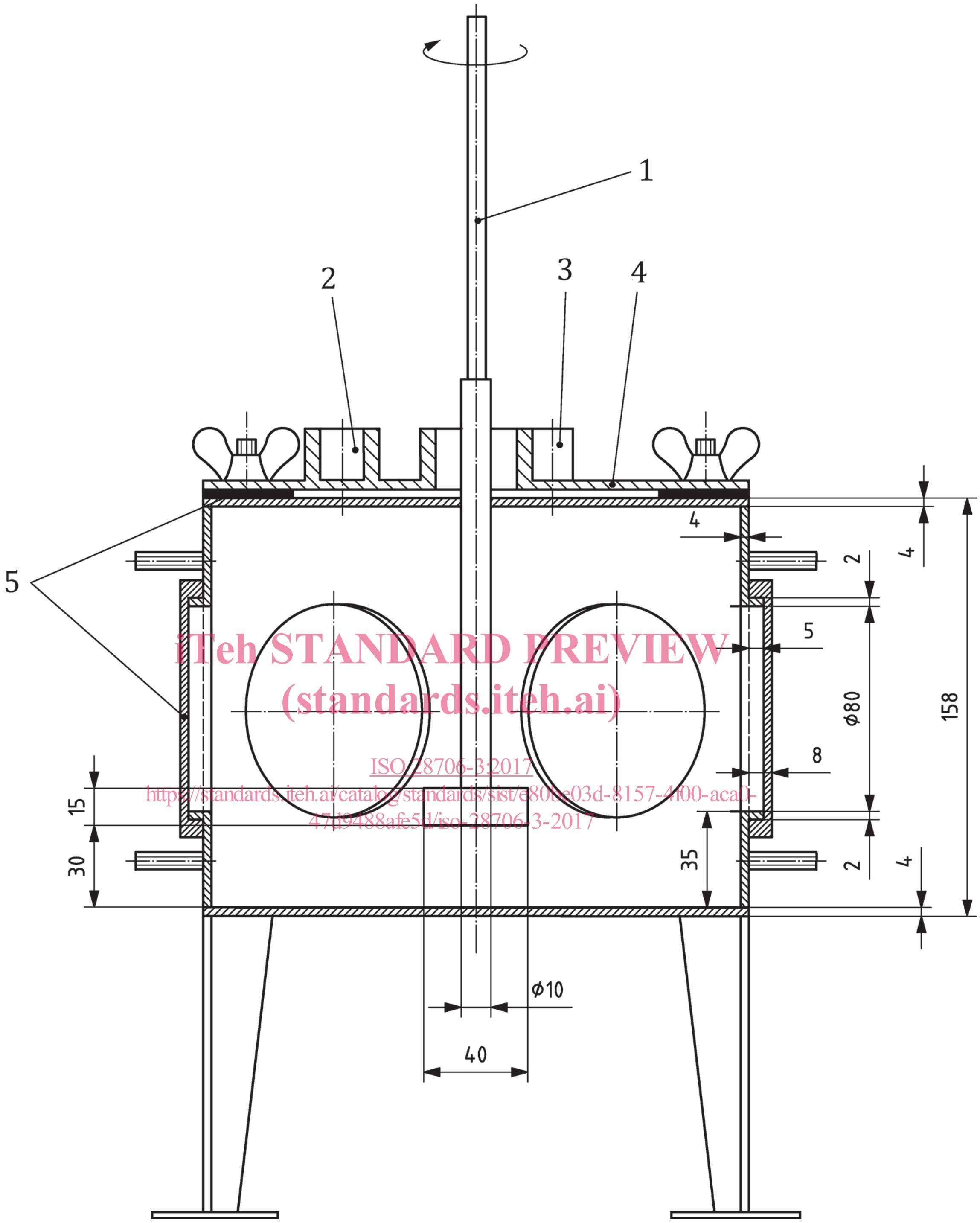


Key

1 gripping plate

Figure 2 — Top view of hexagonal vessel without lid and paddle stirrer

Dimensions in millimetres



Key

1 paddle stirrer

2 socket for temperature-controlling device

3 socket for immersion heater

4 lid

5 sealing rings

Figure 3 — Section A-A of the hexagonal vessel, lid and paddle stirrer, showing sealing rings

