INTERNATIONAL STANDARD

ISO 28706-5

First edition
2008-12-01

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

Part 5: Determination of resistance to chemical corrosion in closed systems

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

Partie 5: Détermination de la résistance à la corrosion chimique en milieux fermés

Reference number
ISO 28706-5:2008(E)

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Published in Switzerland

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 28706-5 was prepared by the European Committee for Standardization (CEN) (as EN 14483-5) and was adopted, under a special “fast-track procedure”, by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, in parallel with its approval by the ISO member bodies.

It cancels and replaces ISO 13806:1999, which has been technically revised.

ISO 28706 consists of the following parts, under the general title *Vitreous and porcelain enamels — Determination of resistance to chemical corrosion*:

— *Part 1: Determination of resistance to chemical corrosion by acids at room temperature*

— *Part 2: Determination of resistance to chemical corrosion by boiling acids, boiling neutral liquids and/or their vapours*

— *Part 3: Determination of resistance to chemical corrosion by alkaline liquids using a hexagonal vessel*

— *Part 4: Determination of resistance to chemical corrosion by alkaline liquids using a cylindrical vessel*

— *Part 5: Determination of resistance to chemical corrosion in closed systems*

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 hydrolysed 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 Clause 9 of ISO 28706-4:2008) the silica network of the enamel is considerably attacked at 80 °C. Silicates and most of the other hydrolysed 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 Clause 9 of ISO 28706-1:2008) or also in stronger acids like sulfuric acid (see Clause 10 of ISO 28706-1:2008), 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 Clause 10 of ISO 28706-2:2008) and boiling 30 % sulfuric acid (see Clause 11 of ISO 28706-2:2008)

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 Clause 12 of ISO 28706-2:2008)**

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 Clause 8 of ISO 28706-5:2008), simulated test solutions (see Clause 10 of ISO 28706-5:2008) or process fluids (see Clause 11 of ISO 28706-5:2008) 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 Clause 13 of ISO 28706-2:2008), 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 9 of ISO 28706-3:2008), 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 Clause 14 of ISO 28706-2:2008) and other alkaline solutions (see Clause 10 of ISO 28706-3:2008 and Clause 10 of ISO 28706-4:2008), 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 this International Standard.

For vitreous enamels fired at temperatures below 700 °C, the test parameters (media, temperatures and times) of this International Standard 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 Parts 1, 2, 3 and 4 of this International Standard.

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

Part 5: Determination of resistance to chemical corrosion in closed systems

1 Scope

This part of ISO 28706 specifies a test method for the determination of the resistance of vitreous and porcelain enamelled articles to attack in closed systems by acid, neutral and alkaline liquids, as well as by actual process mixes.

It applies primarily to the testing of enamels designed for use in chemical processes.

2 Normative references

The following referenced documents are indispensable for the application 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 649-1, *Laboratory glassware — Density hydrometers for general purposes — Part 1: Specification*

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

3 Principle

Enamelled test specimens are exposed in an autoclave, under defined conditions, to attack by a liquid which is corrosive at temperatures above its normal boiling point.

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

4 Apparatus

4.1 Test vessel

4.1.1 Design

The ratio between the volume, V, of the test solution, in cubic centimetres, at 20 °C, and the exposed area of enamel, A, in square centimetres, shall be $V/A = (40 \pm 2)$ cm. The vessel shall be filled to a level such that, when closed and given an ambient temperature of 18 °C to 28 °C, at least 20 % of its volume remains available as a vapour headspace. To ensure this requirement is met, the size of the test apparatus shall be selected to suit that of the specimen.

NOTE Several enamelled specimens can be placed in the same test vessel and tested simultaneously.

WARNING — The test vessel may be a pressure vessel. Attention is drawn to national and international regulations regarding the safe use of pressure vessels.

4.1.2 Material

The test vessel shall be made of a material resistant to the test solution and not releasing any substances that might influence the corrosion of the enamel. In particular, glass or ceramic flasks and fittings or coatings made of fluorinated plastics shall be avoided. Used as a component of seals, PTFE (polytetrafluorethylene) is the only fluorinated plastic suitable for tests with mineral acids, e.g. sulfuric acid and hydrochloric acid.

NOTE Vessels with tantalum fittings or with electrolytically deposited tantalum coatings and vessels made of solid tantalum meet these requirements for acid and neutral solutions over a wide range of applications. For tests with alkaline liquids, both vessels made of plastic materials, e.g. polypropylene bottles, and vessels made of high-alloy austenitic steel are suitable.

4.1.3 Fittings in the test vessel

Fittings in the test vessel are optional, e.g. the test vessel can be equipped with a protective rod for the temperature probe, a specimen holder and other fittings (e.g. agitator and gas supply hose).

4.1.4 Heating device

The type of heating device and its power rating, especially in the case of tests with pressure vessels and temperatures above the boiling point, shall be selected such that the test temperature is reached within 1 h and controllable to within 1 °C, where the test temperature is defined as the temperature of the test solution at the interface with the enamel surface.

The temperature of the test solution is assumed to be locally constant during the exposure period if the test is carried out in the liquid phase.

At test temperatures lower than 100 °C, and especially when using test vessels made of plastic material (for example polypropylene bottles), a thermostatically controlled bath filled with demineralized or distilled water and fitted with an agitator or circulation pump shall be used. This may hold one or more test vessels. The bath shall be covered to avoid loss of liquid by evaporation and shall be capable of maintaining the temperature constant to within 0,1 °C up to 100 °C.

4.2 Analytical balance, capable of weighing to ± 0,02 mg.

4.3 Oven, capable of maintaining a temperature of at least 120 °C.

4.4 Desiccator, capable of holding the test specimens.

4.5 Sponge or **cotton wool**, for cleaning the test specimens.

4.6 Graduated hydrometer, conforming to the requirements of ISO 649-1.

5 Test specimens

5.1 Shape and preparation

The enamel coating on the test specimens shall cover them completely and be free from pinholes. The base metal and the process used to shape the test specimens shall be selected such that there is no risk of localized corrosion occurring as a result of edge spalling or burn marks.

The composition of the enamel on the test specimens and the process by which it is applied shall be the same as in the production process for which testing is being carried out.

The total mass of each test specimen shall not exceed 160 g. The ratio between the area of the exposed surface, A, in square centimetres, and the mass, m, in grams, of the test specimen shall be greater than 0,1 cm^2/g.

5.2 Number

At least two test specimens shall be tested, the actual number of test specimens depending on the number of individual values required to calculate the arithmetic mean (see 7.2).

5.3 Cleaning, conditioning and weighing

Degrease the test specimens, rinse them with demineralized water and then dry them in the oven (4.3) for at least 2 h at 110 °C ± 5 °C. Once the test specimens are dry, cool them in the desiccator (4.4) for at least 2 h and weigh them to the nearest 0,02 mg immediately after removal from the desiccator.

6 Procedure

6.1 General procedure

Pour the test solution (see Clauses 8, 10 and 11) into the test vessel so as to immerse completely the surface of the test specimens to be exposed. For safety reasons, the vapour headspace requirements given in 4.1.1 shall be respected.

After closing the test vessel, heat within 1 h to the test temperature (see 8.3, 10.3 and 11.3).

Start the exposure period of 24 h ± 5 min as soon as the test temperature is reached.

At the end of the exposure period, switch off the heating device and allow the test vessel to cool in air.

This procedure may be used both with vessels in which the test specimens are kept under pressure and with vessels in which the test specimens are at atmospheric pressure.

6.2 Special procedure for plastic bottles

If the test solution is alkaline (see Clause 9) or if the test temperature does not exceed 100 °C or is always below the boiling point of the test solution, tests may be carried out using plastic bottles. Adjust the thermostat of the thermostatically controlled bath to the required test temperature. In the case of hot sodium hydroxide solution, perform all tests as specified in Clause 9.

Fill the thermostatically controlled bath with sufficient water to cover the bottles up to the screw thread. Fill each bottle with test solution to the required depth (see 4.1), close the bottles tightly and place them in the bath set at the test temperature (see 9.3, 10.3 and 11.3). When the test temperature is reached, place each specimen in its bottle. Close the bottle and start the test.

Remove the bottle from the bath after 24 h ± 5 min.

6.3 Washing, drying and weighing of exposed test specimens

Remove the test specimens from their test vessels and wash them with a sponge or with cotton wool (4.5) and demineralized water. Remove any reaction products still adhering with mild, non-abrasive cleaning agents.

Care shall be taken to ensure that the cleaning procedure does not damage the enamel, e.g. by scratching it.