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

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

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*Émaux vitrifiés — Détermination de la résistance à la corrosion
chimique* — 28706-4:2016

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*Partie 4: Détermination de la résistance à la corrosion chimique par
des liquides alcalins dans un récipient cylindrique*



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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.

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 meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#).

The committee responsible for this document is ISO/TC 107, *Metallic and other inorganic coatings*.

This third edition cancels and replaces the second edition (ISO 28706-4:2008), of which it constitutes a minor revision.

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 ($\text{g/m}^2\cdot\text{h}$) 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 this part of ISO 28706), 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 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 of ISO 28706-2:2008, Clause 10) **and boiling 30 % sulfuric acid** (see ISO 28706-2:2008, Clause 11)

Since only minor 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 28706-2:2008, Clause 12)

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 28706-2:2008, Clause 13), 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 ISO 28706-3:2008, Clause 9), 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 28706-2:2008, Clause 14) and other alkaline solutions (see ISO 28706-3:2008, Clause 10 and [Clause 11](#) of this part of ISO 28706); 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 part of ISO 28706 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 4:

Determination of resistance to chemical corrosion by alkaline liquids using a cylindrical vessel

WARNING — This document calls for the use of substances and/or procedures that may be injurious to health if adequate safety measures are not taken. This document does not address any health hazards, safety or environmental matters associated with its use. It is the responsibility of the user of this document to establish appropriate health, safety and environmentally acceptable practices and take suitable actions for any national and International regulations. Compliance with this document does not of itself confer immunity from legal obligations.

1 Scope

This part of ISO 28706 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 cylindrical vessel in which only one enamelled specimen is tested.

NOTE 1 The test method was initially set up for determination of the resistance of vitreous and porcelain enamels to a hot sodium hydroxide solution. Within the scope of this part of ISO 28706, the resistance of other alkaline liquids can be tested.

NOTE 2 This part of ISO 28706, which uses a cylindrical vessel, is generally used for tests carried out on vitreous and porcelain enamel coatings for the chemical industry.

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

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

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

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

EN 10088-1, *Stainless steels — Part 1: List of stainless steels*

3 Principle

An enamelled specimen is exposed to attack by an alkaline liquid under specified conditions of temperature and time. The solution is not stirred during the test.

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

4 Reagents

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

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

4.2 Acetic acid solution, 50 ml/l, for cleaning the test specimens.

4.3 Degreasing solvent, such as ethanol (C₂H₅OH), or water containing a few drops of liquid detergent, suitable for cleaning and degreasing the test specimens.

4.4 Sodium hydroxide (NaOH).

5 Apparatus and material

5.1 Test apparatus: cylindrical vessel.

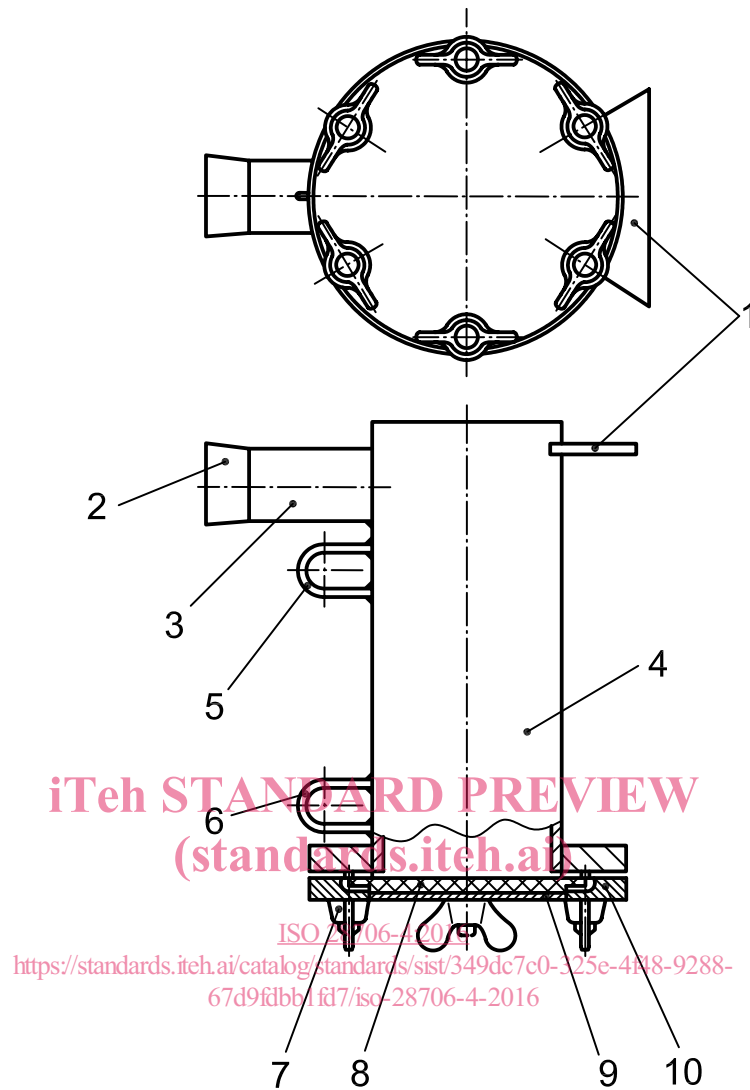
5.1.1 General

The assembled test apparatus is shown in [Figure 1](#). It is composed of the cylinder with the following welded-on elements as shown in [Figure 2](#):

- a plate on one end;
- a circular flange with six welded-on threaded bolts at the other end;
- a foot at the end remote from the circular flange;
- two lifting rings;
- a filling nozzle.

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**Key**

1	foot	6	lifting ring
2	stopper	7	wing nut
3	filling nozzle	8	test specimen
4	cylinder	9	protective envelope
5	lifting ring	10	flange plate

Figure 1 — Test apparatus

The cylinder is sealed by the flange plate and a specimen which is enclosed in a protective envelope and put between the cylinder and the flange plate. The flange plate is fastened to the circular flange by means of six wing nuts. The filling nozzle is closed with a stopper.

The height of the foot depends on the outer diameter of the circular flange. It shall be placed in a way that the surface of the test specimen is totally covered by the liquid if the test apparatus is filled with 1 l of the test solution and placed on a plane surface.

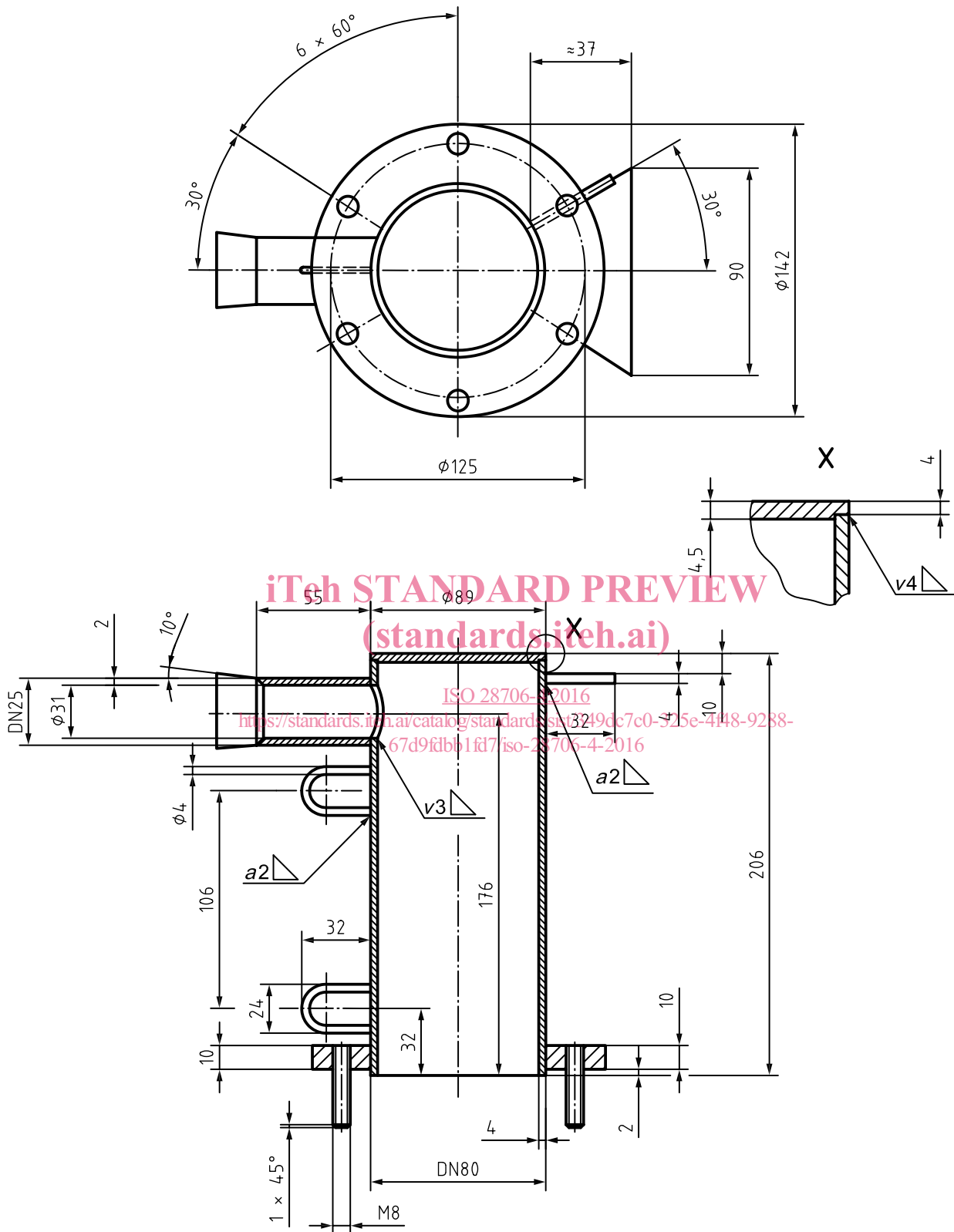


Figure 2 — Cylinder

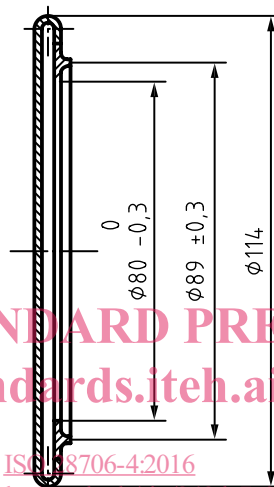
5.1.2 Cylinder, with all surfaces consisting of bare metal and all edges deburred. All parts shall be made of the same stainless steel, for example 1.4571 stainless steel conforming to the requirements of EN 10088-1.

NOTE The main constituents of 1.4571 steel are 16,5 % to 18,5 % Cr, 10,5 % to 13,5 % Ni, 2 % to 2,5 % Mo, maximum 0,08 % C and an addition of Ti.

5.1.3 Protective envelope, shown in [Figure 3](#), made of synthetic rubber of hardness 70 IRHD as determined in accordance with ISO 48. The material shall be resistant to alkaline solutions at 100 °C (e.g. chloroprene or ethylene-propylene).

The rubber and the circular dam usually have a thickness of 2 mm; other thicknesses can also be used, however.

Dimensions in millimetres



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Figure 3 — Protective envelope

5.1.4 Flange plate, shown in [Figure 4](#), with all surfaces consisting of bare metal and all edges deburred. It shall be made of stainless steel, for example 1.4571 stainless steel conforming to EN 10088-1.