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

Part 2:

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

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

*Partie 2: Détermination de la résistance à la corrosion chimique par des
acides bouillants ou des liquides neutres bouillants, et/ou leurs vapeurs*



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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 28706-2 was prepared by the European Committee for Standardization (CEN) (as EN 14483-2) 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 2733:1983, ISO 2742:1998, ISO 2743:1986 and ISO 2744:1998, which have 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 ($\text{g/m}^2\cdot\text{h}$) 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 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 28706-2:2008), 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), artificial 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" of Parts 1, 2, 3 and 4 of this International Standard.

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

Part 2:

Determination of resistance to chemical corrosion by boiling acids, boiling neutral liquids and/or their vapours

1 Scope

This part of ISO 28706 describes a test method for the determination of the resistance of flat surfaces of vitreous and porcelain enamels to boiling acids, boiling neutral liquids and/or their vapours.

This method allows the determination of the resistance of vitreous and porcelain enamels to the liquid and vapour phases of the corrosive medium simultaneously.

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

ISO 649-1, *Laboratory glassware — Density hydrometers for general purposes — Part 1: Specification*

ISO 718, *Laboratory glassware — Thermal shock and thermal shock endurance — Test methods*

ISO 3585, *Borosilicate glass 3.3 — Properties*

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

ISO 4788, *Laboratory glassware — Graduated measuring cylinders*

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 Principle

A set of similarly enamelled test specimens is placed in the liquid zone and/or in the vapour zone of the test apparatus, as required, and exposed to attack by a boiling acid or boiling neutral liquid, or its vapour, under specified conditions.

The same design of test apparatus and the same test principle is employed for the different liquids.

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, volume concentration 50 ml/l, for cleaning the test apparatus and test specimens.

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

4.4 Citric acid monohydrate, ($C_6H_8O_7 \cdot H_2O$), crystalline.

4.5 Sulfuric acid, analytical grade, 30 % (by mass) solution, density range 1,217 g/ml to 1,220 g/ml (measured with a hydrometer — see 5.8).

4.6 Hydrochloric acid, analytical grade, 20 % (by mass) solution, density range 1,097 g/ml to 1,099 g/ml (measured with a hydrometer — see 5.8).

5 Apparatus and materials

5.1 Test apparatus

5.1.1 General description

The test apparatus (see Figures 1 and 2) consists of a cylinder (5.1.2) (see Figure 3), with an adjacent support, having a standard socket for holding a reflux condenser (5.1.3) with a graduated collector (5.1.4) on one side.

Two test specimens shall form the top and bottom of the cylinder. One of them may be replaced by a glass plate (5.1.14) if required. The cylinder with the specimens shall be supported between two plates (see Figure 2) locked at the corners by threaded bolts (5.1.8), wing nuts (5.1.7) and hexagonal nuts (5.1.6). A synthetic-fibre washer (5.1.9) is fixed between the plates (5.1.5) and each specimen. The specimens are sealed against the ends of the cylinder with packing rings (5.1.10), the material of which is dependent on the type of test solution. Any uncoated area of the test specimen shall be protected from exposure to the attacking medium.

When testing specimens cut from an enamelled article, the packing rings (5.1.10) are replaced by protective envelopes (see Figure 5) in which the specimens are placed.

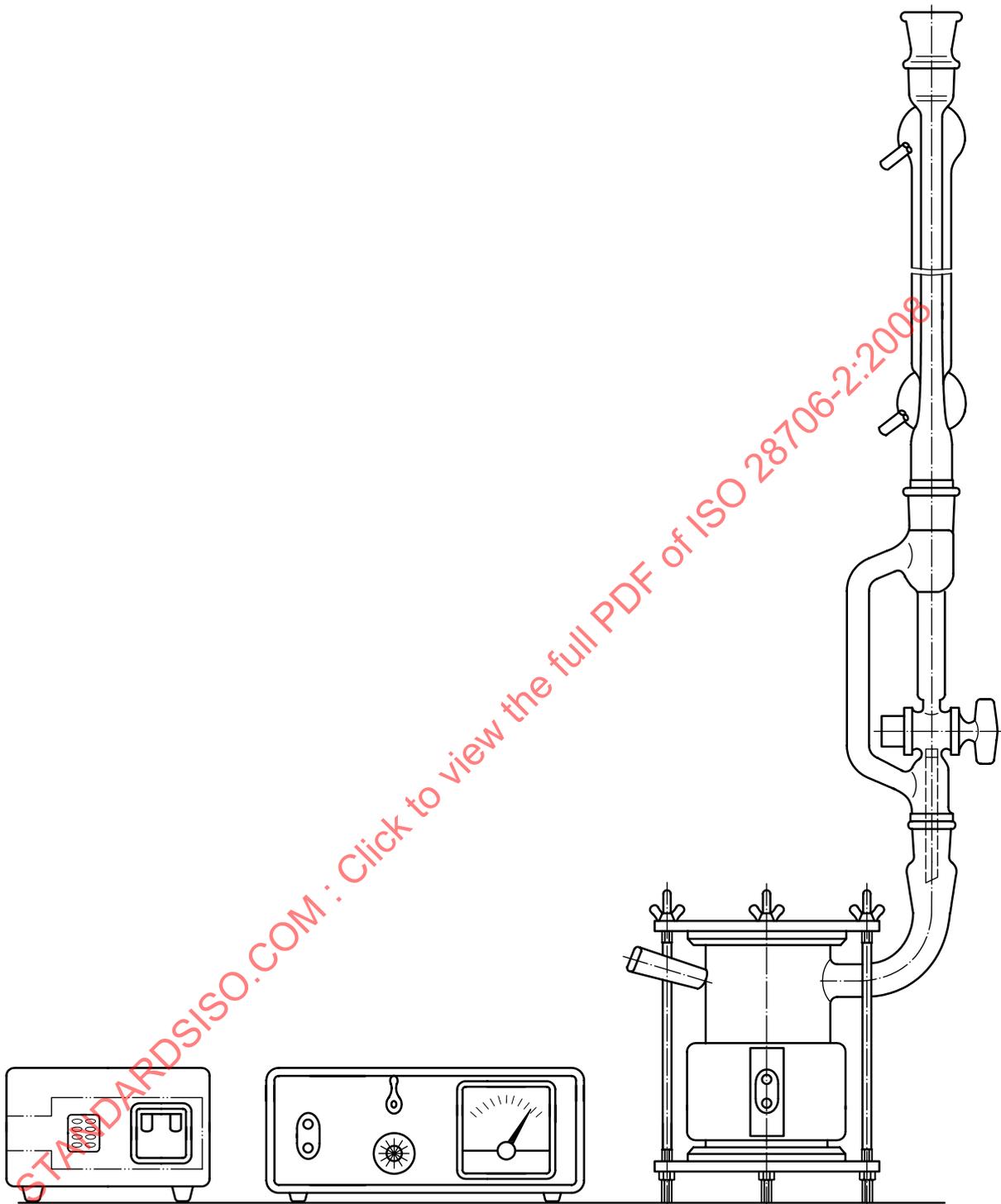
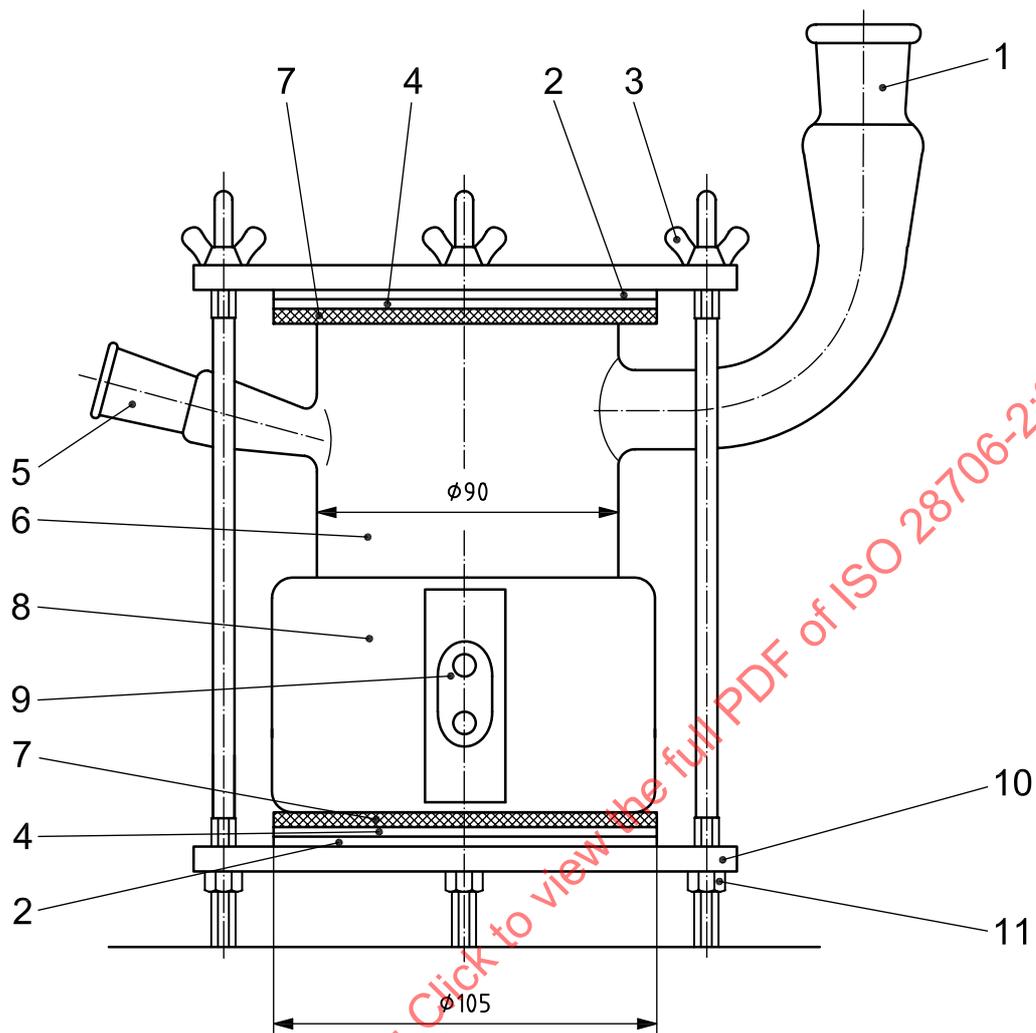


Figure 1 — View of assembled test equipment

Dimensions in millimetres



Key

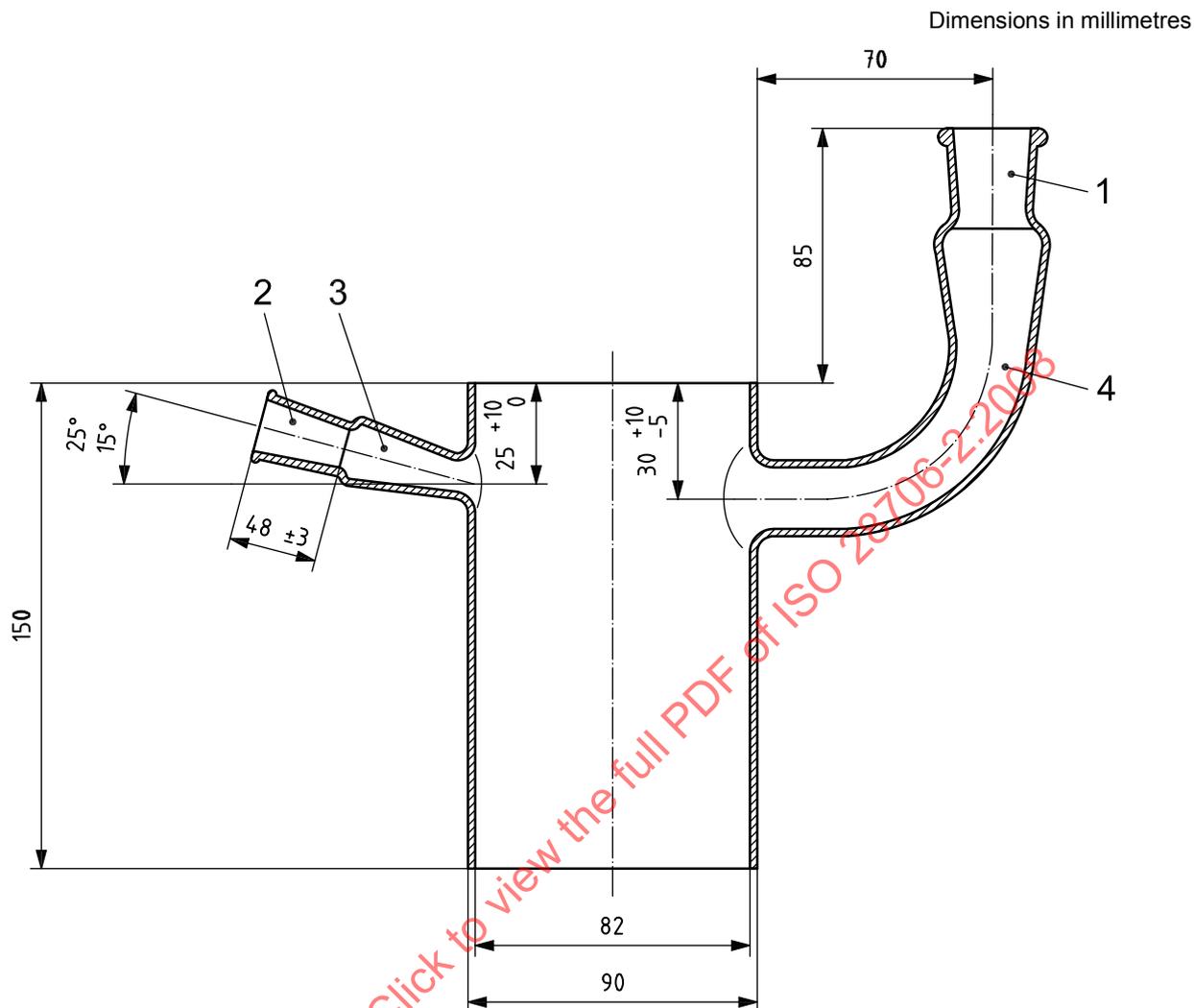
- | | | | |
|---|-----------------------------|----|------------------|
| 1 | socket for reflux condenser | 7 | packing ring |
| 2 | synthetic-fibre washer | 8 | heater |
| 3 | wing nut | 9 | electric socket |
| 4 | specimen | 10 | triangular plate |
| 5 | socket for thermometer | 11 | hexagonal nut |
| 6 | cylinder | | |

Figure 2 — Test apparatus

The apparatus is heated externally by a heater (5.1.11) placed round the lower half of the cylinder (5.1.2) such that the lower edge is, at the most, 3 mm above the lower packing ring. The test apparatus is composed of the following parts:

5.1.2 Cylinder (see Figure 3), made of borosilicate glass 3,3 conforming to the requirements of ISO 3585, with ground ends. When tested in accordance with ISO 718, the cylinder shall pass the test without breaking at a difference in temperature of at least 120 °C.

NOTE Cylinders having two sockets can also be used if the smaller socket is closed by a stopper which is resistant to the boiling solution.



Key

- 1 ground-glass socket for condenser
- 2 ground-glass socket for thermometer
- 3 connecting piece
- 4 connecting piece

Figure 3 — Cylinder

5.1.3 Liebig-West reflux condenser, or equivalent reflux condenser conforming to ISO 4799, in which there is no volume change during the test, with a nominal jacket length of 400 mm and standard ground joint of borosilicate glass 3,3 conforming to the requirements of ISO 3585.

5.1.4 Graduated collector (see Figure 4), with a standard ground joint of borosilicate glass 3,3 conforming to the requirements of ISO 3585, arranged in the apparatus to collect the condensate produced in the reflux condenser. The graduation interval shall be 0,1 ml.

Dimensions in millimetres

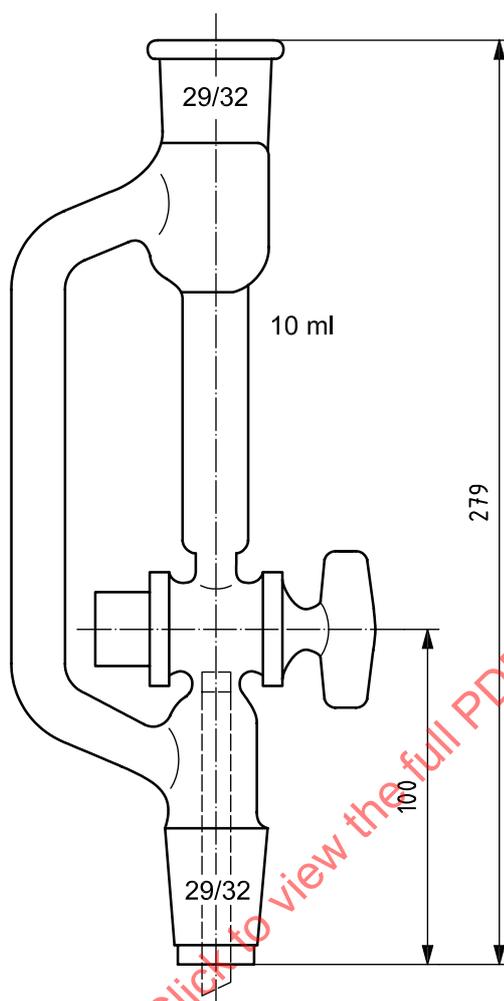


Figure 4 — Graduated collector

Dimensions in millimetres

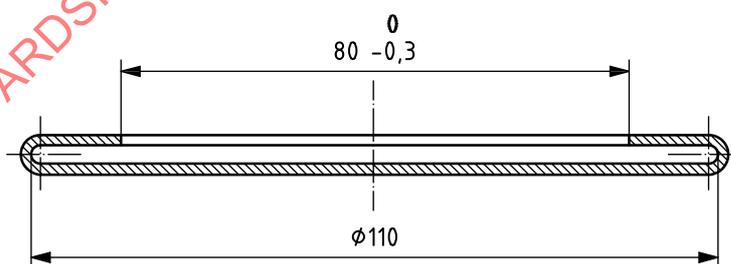


Figure 5 — Protective envelope

- 5.1.5 Two plates of surface-protected steel.
- 5.1.6 Hexagonal nuts, with a thread fitting the screw bolts.
- 5.1.7 Wing nuts, with a thread fitting the screw bolts.

5.1.8 Threaded bolts, of corrosion-resistant steel.

5.1.9 Synthetic-fibre washers, resistant to acid and water at 140 °C.

NOTE PTFE is the only plastic material suitable for tests with mineral acids (e.g. H₂SO₄, HCl).

5.1.10 Packing rings (see Clause 6).

5.1.11 Heater, with an output of 400 W to 500 W, constructed of heat-conducting alloy covered with heat-insulating material, having dimensions such that the lower edge of the heater is, at the most, 3 mm above the lower packing ring and is not in contact with it.

5.1.12 Heat-controlling device, for example a thermostat, a variable transformer or electronic control equipment.

5.1.13 Voltage stabilizer, to avoid variation in heating due to fluctuations in the power supply.

5.1.14 Glass plate, made of borosilicate glass 3,3 conforming to the requirements of ISO 3585, having a diameter of 105 mm, as cover or bottom of the cylinder, if required.

5.1.15 Simmering aid, such as floating particles resistant to the boiling solution.

NOTE 1 PTFE is the only fluorinated plastic material suitable for tests with mineral acids (e.g. H₂SO₄, HCl).

NOTE 2 When boiling with sulfuric acid, boiling retardation is best avoided by using borosilicate glass capillary tubes.

5.2 Drying oven, capable of maintaining a temperature of at least 130 °C.

5.3 Desiccator, for example with an internal diameter of 200 mm.

5.4 Graduated measuring cylinder, capacity 500 ml, conforming to the requirements of ISO 4788.

5.5 Beakers.

5.6 Balance, capable of weighing to the nearest 0,2 mg.

5.7 Sponge, soft.

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

6 Packing rings

6.1 General

The method of holding the specimens against the ends of the cylinder depends on the type of specimen and the type of test solution. Use one of the following types of packing:

6.2 Packing A

Compressed-fibre washers 100 mm in external diameter, 80 mm ± 1 mm in internal diameter and 2 mm thick, covered with a plastic material resistant to hydrochloric acid at 140 °C (for example polytetrafluorethene).

6.3 Packing B

Packing rings 100 mm in external diameter, 80 mm \pm 0,3 mm in internal diameter and 2 mm or 3 mm thick, made of rubber with a hardness of 70 IRHD as determined in accordance with ISO 48 and resistant to citric acid and water at 140 °C (for example chloroprene or ethylene-propylene).

7 Test specimens

Prepare the test specimens in accordance with ISO 28764.

Rinse each test specimen with water (4.1). If necessary, use a suitable degreasing solvent (4.3). Dry each specimen for 2 h in the drying oven (5.2) maintained at 110 °C \pm 5 °C. Allow the specimens to stand for at least 2 h in the desiccator (5.3) and finally weigh each to the nearest 0,2 mg. Record the starting mass, m_s .

8 Procedure

Carry out two determinations, with new test specimens for each determination, in vapour and/or liquid.

Fix the test specimens in the test apparatus (5.1) so that the coated faces of the specimens are facing the interior of the cylinder. Protect any uncoated areas of the test specimens from exposure to the attacking medium. If the test is to be carried out only in the liquid phase, use a glass plate of borosilicate glass 3.3 (ISO 3585) as the cover for the apparatus; if the test is to be carried out only in the vapour phase, use a glass plate of borosilicate 3.3 (ISO 3585) as the bottom of the apparatus.

Screw down the wing nuts evenly to ensure that the test apparatus is tight to liquids.

Run 450 ml of the test solution (see Clauses 10 to 13) into the cylinder through the socket provided for the graduated collector and mount the latter and the reflux condenser in place. Switch the heater on, and bring the test solution to boiling within 15 min. The measured test period starts with the commencement of boiling. As soon as the solution is boiling vigorously, use the heat-controlling device to maintain the amount of condensate condensed in the reflux condenser and measured in the graduated collector within the range 8 ml \pm 2 ml every 3 min for the duration of the test.

The duration of the test depends on the test solution (see Clauses 10 to 13).

After heating for the prescribed period (see Clauses 10 to 13), empty the cylinder, cool it and rinse it with water (4.1).

Take the specimens from the apparatus, wipe them three times with a sponge (5.7) which has been soaked in acetic acid (4.2) at room temperature and then rinse with water (4.1).

After carefully removing any packing-ring residues from the edges of the specimens, dry them for 2 h in the hot-air oven (5.2) at 110 °C \pm 5 °C. After a further 2 h in the desiccator (5.3), weigh each specimen again to the nearest 0,2 mg and record the final mass, m_f . The total time from removal of a specimen from the desiccator until completion of weighing shall not exceed 2 min.

Measure the diameter of the area exposed to attack. The mean value of three measurements of the diameter of the area exposed to attack shall lie within \pm 1 mm of 80 mm. Calculate the area exposed to attack, A , using this mean value of the diameter.

9 Expression of results

9.1 Total loss in mass per unit area

For each test, calculate the total loss in mass per unit area, $\Delta\rho_A$, in g/m^2 , for the total duration of the test, from the equation:

$$\Delta\rho_A = \frac{(m_s - m_f)}{A} \quad (1)$$

where

m_s is the starting mass, in g;

m_f is the final mass, in g;

A is the area exposed to attack, in m^2 .

In order to distinguish between test results for different test periods, the number of test hours shall be stated as a subscript to the symbol, for example:

for a test period of 2,5 h: $\Delta\rho_{A2,5}$

for a test period of 48 h: $\Delta\rho_{A48}$

Test specimens which show defects such as pinholes down to the metal, chipped edges or edge corrosion shall be discarded and a corresponding number of new specimens shall be tested.

Express the result as the arithmetic mean of the individual values to the nearest $0,1 \text{ g/m}^2$. The individual values shall not differ from the mean value by more than 20 %.

9.2 Corrosion rate

In the hydrochloric acid test (see Clause 12), the corrosion of the enamel proceeds linearly with time. The corrosion rate, v , expressed as the rate of loss in mass per unit area, $\text{g}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$, is calculated from the equation:

$$v = \frac{\Delta\rho_A}{t} \quad (2)$$

where t is the test time, in h.

Calculate the corrosion rate, w , in mm per year from the equation:

$$w = 3,504v \quad (3)$$

NOTE In Equation (3), it is assumed that enamel is a homogeneous material (without gas bubbles) with a density of $2,5 \text{ g/cm}^3$.

Express the result as the arithmetic mean of the individual values, in millimetres per year, to the nearest $0,01 \text{ mm/year}$.

10 Boiling citric acid

10.1 General

Carry out this test using the procedure described in Clause 8.

10.2 Citric acid test solution

Dissolve 32 g of pure crystalline citric acid monohydrate ($C_6H_8O_7 \cdot H_2O$) in 500 ml of water (4.1). A fresh solution, prepared the same day, is required for each test.

Only the resistance to chemical corrosion in the liquid phase shall be assessed.

10.3 Duration of the test

The heating time, i.e. the time with the condensation rate maintained within the range $8 \text{ ml} \pm 2 \text{ ml}$ per 3 min, shall be 2,5 h (150 min).

If the loss in mass of any of the specimens after this time is less than 8 mg, the result of the test is " $< 1,6 \text{ g/m}^2$ ".

If a more precise result is required, carry out another test using new specimens and another test solution and/or another test duration after testing with other test solutions and/or conditions as described in Clause 14.

10.4 Test report

The test report shall include the following information:

- a) all information necessary for the identification of the sample tested;
- b) a reference to Clause 10 of this part of ISO 28706, e.g. "Tested in accordance with Clause 10 of ISO 28706-2:2008 — boiling citric acid";
- c) the duration of the test: 2,5 h;
- d) the results, giving the loss in mass per unit area as calculated for the time period over which testing was conducted (see 9.1), in grams per square metre, for each individual determination, plus the arithmetic mean, rounded to the nearest $0,1 \text{ g/m}^2$;
- e) any deviations from the procedure specified;
- f) any unusual features observed during the test;
- g) the date of the test.

11 Boiling sulfuric acid

11.1 General

Carry out this test following the procedure described in Clause 8.

11.2 Test solution

Sulfuric acid, analytical grade, 30 % (by mass) solution, density range $1,217 \text{ g/ml}$ to $1,220 \text{ g/ml}$ (measured with a hydrometer — see 5.8). A fresh solution is required for each test.

Only the resistance to chemical corrosion in the liquid phase shall be assessed.

11.3 Duration of the test

The heating time, i.e. the time with the condensation rate maintained within the range $8 \text{ ml} \pm 2 \text{ ml}$ per 3 min, shall be 18 h.

If the loss in mass of any of the specimens after this time is less than 8 mg, the result of the test is " $< 1,6 \text{ g/m}^2$ ".

If a more precise result is required, carry out another test using new specimens and another test solution and/or another test duration after testing with other test solutions and/or conditions as described in Clause 14.

11.4 Test report

The test report shall include the following information:

- a) all information necessary for the identification of the sample tested;
- b) a reference to Clause 11 of this part of ISO 28706, e.g. "Tested in accordance with Clause 11 of ISO 28706-2:2008 — boiling sulfuric acid";
- c) the duration of the test: 18 h;
- d) the results, giving the loss in mass per unit area as calculated for the time period over which testing was conducted (see 9.1), in grams per square metre, for each individual determination, plus the arithmetic mean, rounded to the nearest $0,1 \text{ g/m}^2$;
- e) any deviations from the procedure specified;
- f) any unusual features observed during the test;
- g) the date of the test.

12 Boiling hydrochloric acid

12.1 General

Carry out this test using the procedure described in Clause 8.

12.2 Test solution

Hydrochloric acid, analytical grade, 20 % (by mass) solution, density range 1,097 g/ml to 1,099 g/ml (measured with a hydrometer — see 5.8). A fresh solution is required for each test.

Only the resistance to chemical corrosion in the vapour phase shall be assessed.

12.3 Duration of the test

The heating time, i.e. the time with the condensation rate maintained within the range $8 \text{ ml} \pm 2 \text{ ml}$ per 3 min, shall be 7 days.

If the loss in mass of any of the specimens after this time is less than 8 mg, carry out the test with new specimens and a heating time of 14 days. If the loss in mass is still less than 8 mg, the result of the test is " $< 1,6 \text{ g/m}^2$ ".

If a more precise result is required, carry out another test using new specimens and another test solution and/or another test duration after testing with other test solutions and/or conditions as described in Clause 14.