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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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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).

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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.

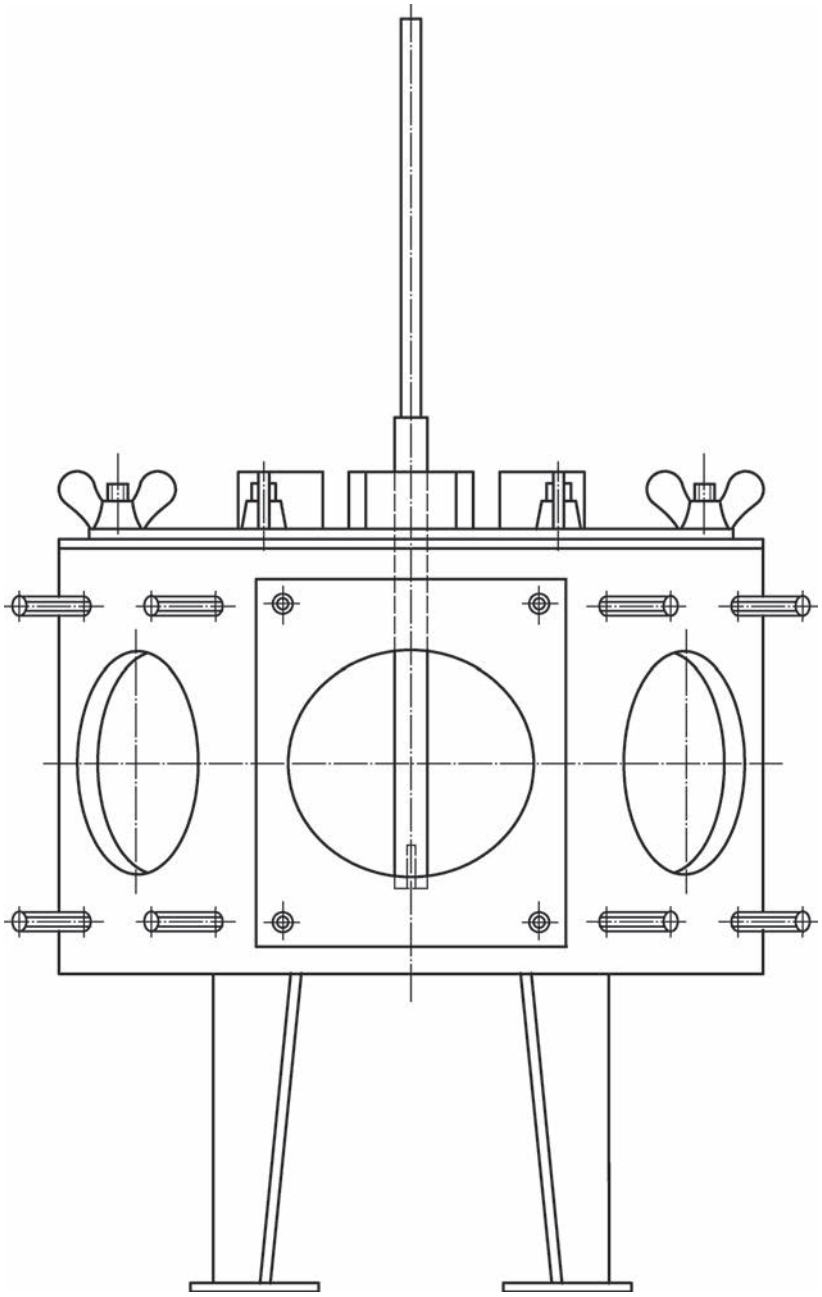
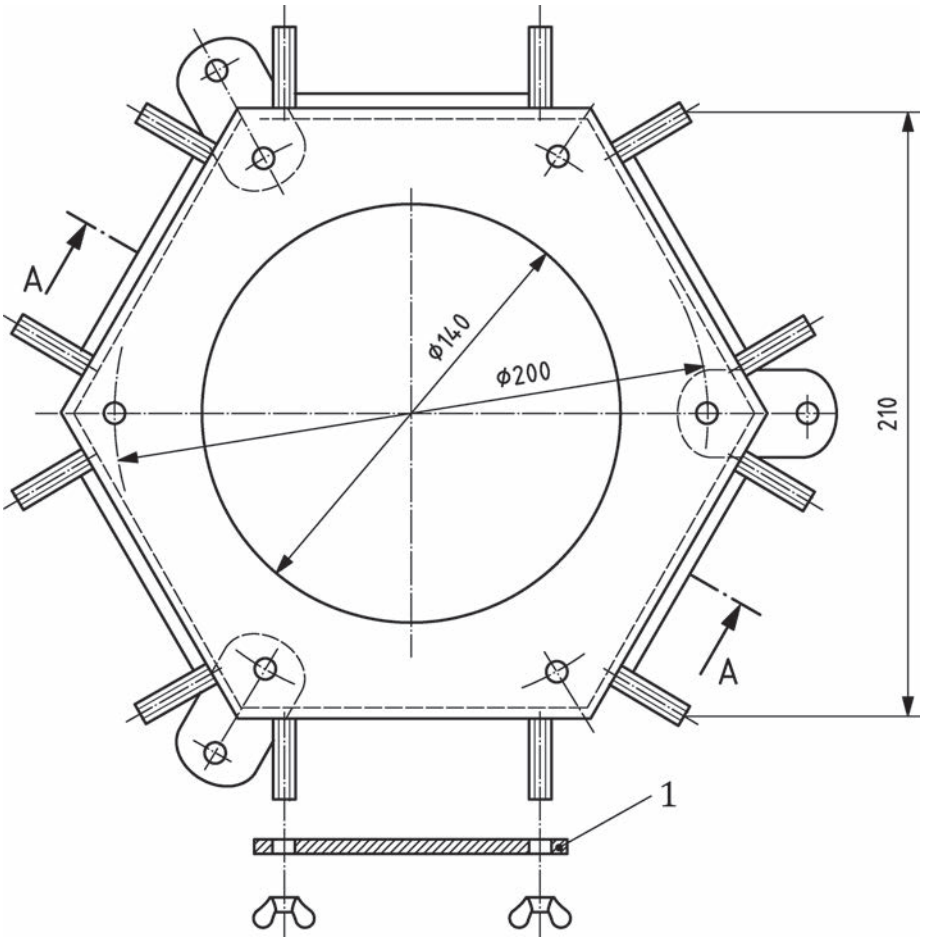


Figure 1 — Hexagonal vessel with lid, stirrer and gripping plate

Dimensions in millimetres

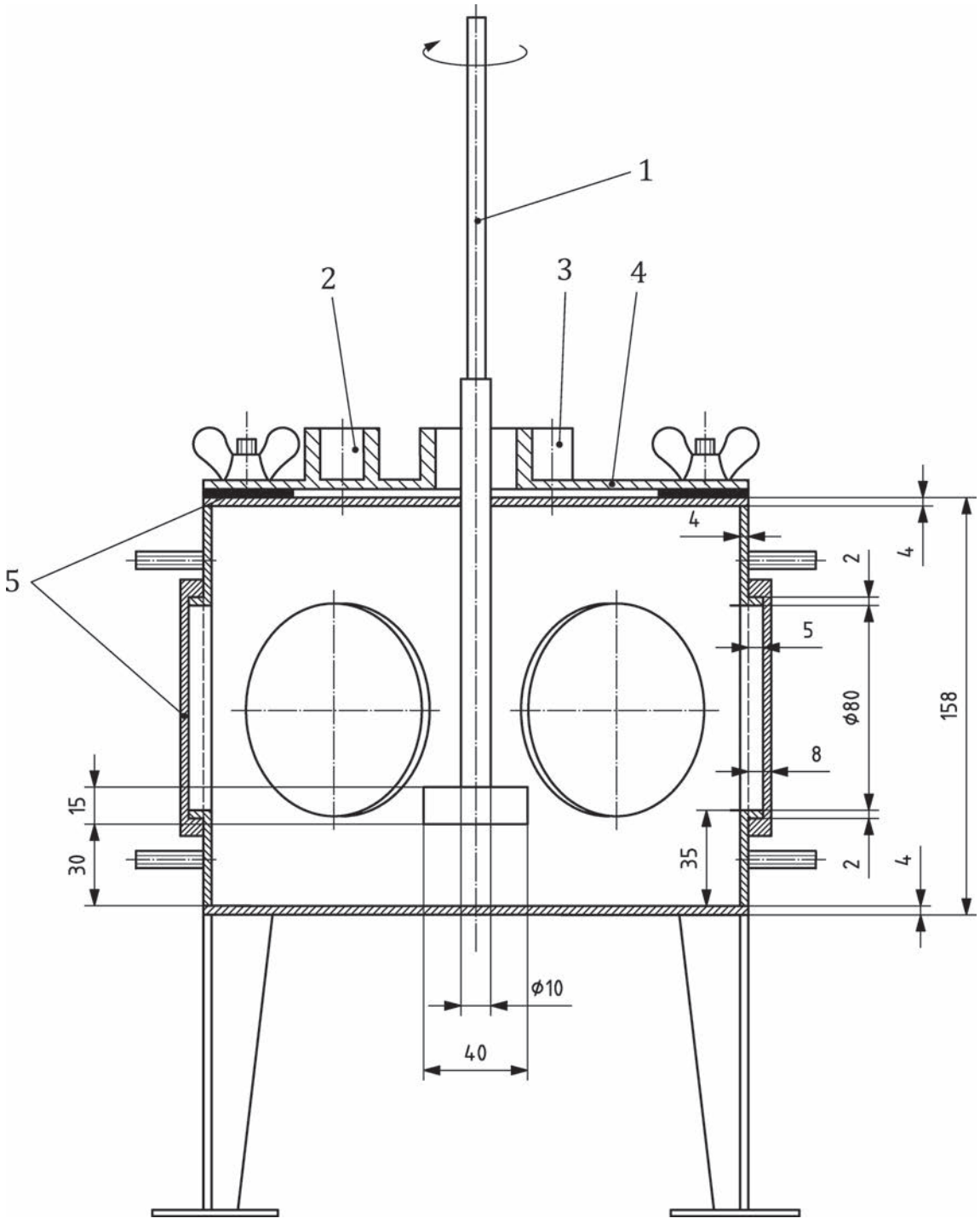


Key

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

Dimensions in millimetres

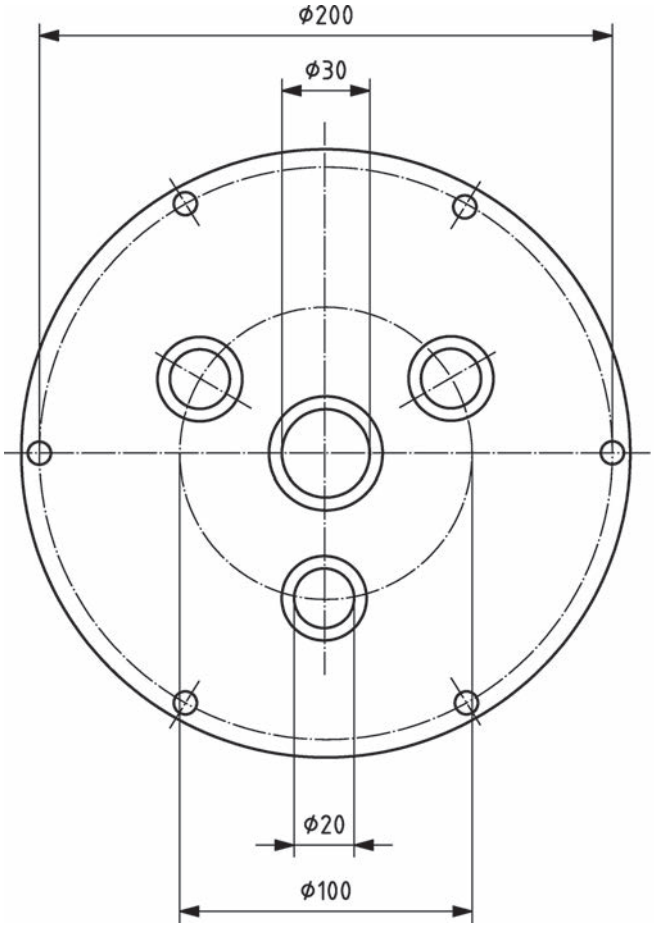


Figure 4 — Top view of lid

The sealing rings (6.1.6) shall be 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 (chloroprene, for example, is suitable).

The hexagonal vessel, lid, gripping plates and paddle stirrer shall be made of the same austenitic stainless steel.

6.1.2 Hexagonal vessel, of austenitic stainless steel (see Figures 1 to 3), with four threaded bolts welded to each side for fastening the gripping plates and with six threaded bolts welded to the upper surface for fastening the lid. The vessel should preferably have an outlet for drainage.

6.1.3 Lid, of austenitic stainless steel, with a centrally placed support for receiving the paddle stirrer and with three further supports for receiving the immersion heaters and the temperature-controlling device.

6.1.4 Gripping plates (six), of austenitic stainless steel of thickness 4 mm, which can be fitted to the sides of the hexagonal vessel.

6.1.5 Wing nuts (30), for fastening the gripping plates and the lid to the vessel.

6.1.6 Sealing rings (six), of external diameter 100 mm, internal diameter 80 mm and thickness 8 mm, for sealing the side openings. An additional ring, of internal diameter 140 mm and of thickness 3 mm, is required to serve as an intermediate layer between the lid and the vessel.

6.1.7 Paddle stirrer, of austenitic stainless steel, with the dimensions shown in Figure 3. The stirrer shall operate at a rotational frequency of (1 350 ± 50) min^{-1}.

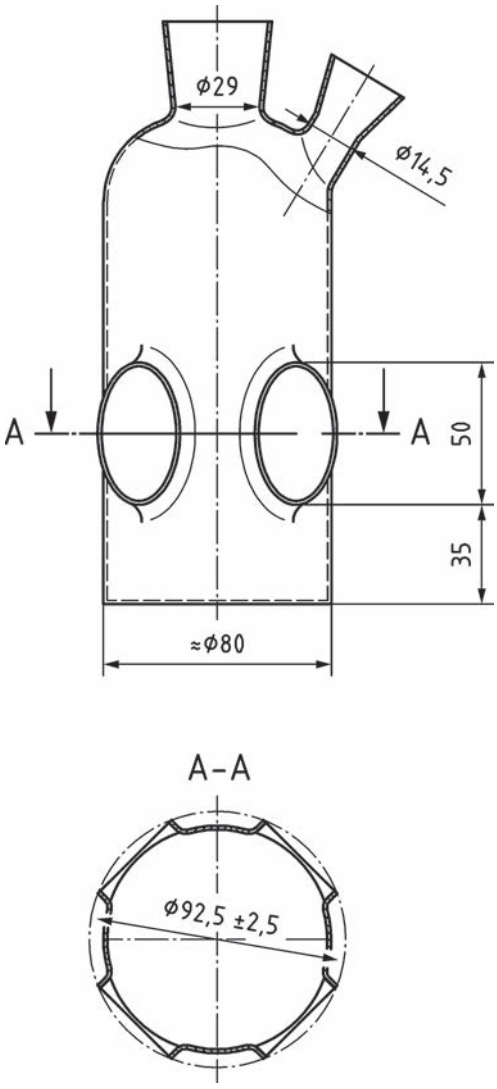
6.1.8 Immersion heaters (two), cylindrical, each of 600 W, made of nickel-plated copper or of austenitic stainless steel.

6.1.9 Temperature-controlling device, comprising a contact thermometer with a thermostat accurate to ±1 °C. The use of a temperature-recording instrument is recommended.

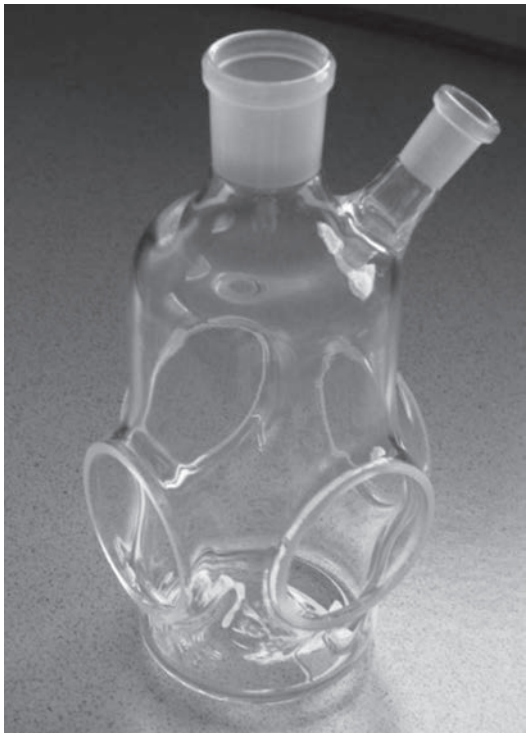
6.2 Tetragonal glass bottle apparatus.

6.2.1 The apparatus (see Figure 5) consists of a tetragonal vessel having a circular opening in 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.

Dimensions in millimetres



a) Drawing of glass bottle



b) Picture of glass bottle

Figure 5 — Tetragonal glass bottle

The bottle of borosilicate glass 3.3 conforming to the requirements of ISO 3585 has two sockets: one used for a cooler, the second (NS 29) for a temperature-controlling device. The sealing rings (6.1.6) shall be 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 (chloroprene, for example, is suitable).

A ring with gripping plates, as shown in Figure 6, is used to keep the test specimens in position. The bottle is heated with a safety magnetic stirrer with heating, suitable for unsupervised operation. The magnetic stirrer shall have a bushing for connecting a contact thermometer which enables precise temperature control (±1 °C). The thermocouple needs a ground glass joint with sleeve coupling to be attached to the apparatus (see Figure 7).

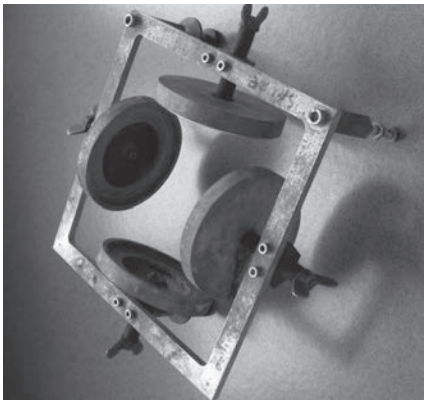


Figure 6 — Ring with gripping plates

Figure 7 — Ground glass joint with sleeve coupling

6.2.2 Magnetic stirrer with heating

6.2.3 Contact thermometer, capable of maintaining a temperature of (100 ± 1) °C.

6.2.4 Ground glass joint, with sleeve coupling for the contact thermometer.

6.2.5 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.

6.2.6 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.

6.2.7 PTFE coated magnetic agitator, to put in the bottle.

6.3 Materials.

6.3.1 Hot-air oven, capable of being maintained at (120 ± 5) °C.

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

6.3.3 Balance, accurate to 0,2 mg.

6.3.4 Cotton wool.

6.3.5 Sponge, soft.

7 Test specimens

The specimens to be used shall be prepared in accordance with ISO 28764. Enamel both sides of the specimens.

8 Procedure

8.1 General

For each determination, two tests with two similarly enamelled specimens shall be carried out.

Before the test, wipe each specimen with cotton wool (6.3.4) soaked in the degreasing solvent (5.2). Then dry the specimens for 2 h in the hot-air oven (6.3.1), controlled at (120 ± 5) °C, cool for at least 2 h in the desiccator (6.3.2) and weigh to the nearest 0,2 mg. Record the starting mass, m_S.

8.2 Hexagonal vessel

Press the specimens against the side openings of the hexagonal vessel and secure them by means of the gripping plates so that the vessel is watertight. Pour 4,5 l of the alkaline test solution (see Clauses 10 and 11), at room temperature, into the vessel through the inlet in the lid. Heat the test solution to the specified temperature (see Clauses 10 and 11), stirring continuously, and maintain it at this temperature for the specified time (see Clauses 10 and 11).

At the end of the required time (see Clauses 10 and 11), remove the hot test solution and fill the vessel immediately with water (5.1) at room temperature. Stir the water (5.1) for 2 min and then remove it. Remove the specimens from the vessel and rinse the vessel thoroughly once more.

Wipe both sides of the specimens with cotton wool soaked in water (5.1) and then rinse with the degreasing solvent (5.2). Dry the specimens for 2 h in the hot-air oven (6.3.1), controlled at (120 ± 5) °C, and then leave them in the desiccator (6.3.2) for 2 h. Weigh each specimen to the nearest 0,2 mg and record the final mass, m_f.

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 ±1 mm of 80 mm. Calculate the area exposed to attack, A, using this mean value of the diameter.

8.3 Tetragonal glass bottle

Fix the test specimens in the test apparatus (6.2) 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.

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

Run 600 ml (see Clauses 10 and 11) of the test solution into the cylinder through the socket provided for the graduated collector and mount the latter and the reflux condenser in place, leaving the graduated collector open. Heat the test solution to the specified temperature (see Clauses 10 and 11). The measured test period starts once the test temperature has been reached. Use the heat-controlling device to maintain the temperature.

After heating for the prescribed period, empty the cylinder, cool it and rinse it with water (5.1).

Take the specimens from the apparatus, wipe them three times with a sponge (6.3.5) which has been soaked in acetic acid (5.8) at room temperature and then rinse with water (5.1).

After carefully removing any packing-ring residues from the edges of the specimens, dry them for 2 h in the hot-air oven (6.3.1) at 110 °C ± 5 °C. After a further 2 h in the desiccator (6.3.2), weigh each specimen again to the nearest 0,2 mg and record the final mass, m_f.

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 ±1 mm of 50 mm. Calculate the area exposed to attack, A, using this mean value of the diameter.

9 Expression of results

For each test, calculate the result as the total loss in mass per unit area, $\Delta\rho_A$, in g/m^2, for the total duration of the test, using Formula (1):

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

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 the 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 24 h, $\Delta\rho_{A24}$.

Results for 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 g/m^2. The individual values shall not differ from the mean value by more than 20 %.

10 Standard detergent solution test

10.1 General

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

10.2 Test solution

Prepare 4,5 l of a solution containing the following:

— 27,0 g of sodium tripolyphosphate ($Na_5P_3O_{10}$);

— 9,0 g of anhydrous sodium carbonate (Na_2CO_3);

— 2,7 g of hydrated sodium perborate ($NaBO_2{\cdot}H_2O_2{\cdot}3H_2O$);

— 1,8 g of sodium silicate, containing about 81 % (by mass) of Na_2SiO_3;

— 4,5 g of alkylsulfonate [$CH_3(CH_2)_x - C(SO_2Na)\ H - (CH_2)_3 - CH_3$].

When using a 600 ml solution, ensure that the proportionate amounts of the above are used.

The solution shall be made up using water (5.1) and reagents of analytical grade.

Use a fresh test solution for each test.

10.3 Test temperature

The test solution shall be heated in the vessel to 95 °C ± 2 °C and shall be maintained at that temperature for the duration of the test.

10.4 Duration of the test

The heating time at 95 °C (i.e. without the heating-up time) shall be 24 h (6.1) and 2,5 h (6.2), respectively.

If the average loss in mass per unit area is less than 8 mg after 24 h, repeat the test with new sets of specimens, increasing the test period to 168 h (only 6.1). Replace the standard test solution after each 24 h period by removing the hot test solution and filling the vessel immediately with fresh test solution at room temperature (only 6.1).

If the loss in mass is still less than 8 mg, the result of the test is "<1,6 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 11.

10.5 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 document, e.g. "Tested in accordance with ISO 28706-3:2017, Clause 10";

c) the duration of the test, in hours, and type of apparatus;

d) the results, giving the loss in mass per unit area as calculated for the time period over which testing was conducted (see Clause 9), in grams per square metre, for each individual determination, plus the arithmetic mean, rounded to the nearest 0,1 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 Other test solutions and/or conditions

11.1 General

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

11.2 Test solution

Prepare the necessary amount of an agreed alkaline test solution, using water (5.1) and reagents of analytical grade. No test solutions shall be used that could damage the apparatus.

Use a fresh test solution for each test.

11.3 Test temperature

The test solution shall be heated in the vessel to an agreed temperature between 40 °C and 95 °C, and shall be maintained at that temperature during the duration of the test.

11.4 Duration of the test

The heating time at the test temperature shall be included in the test report.

If the average loss in mass per unit area is less than 8 mg after this period, the result of the test is "<1,6 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.

If the test time is more than 24 h, the test solution shall be replaced every 24 h by removing the hot standard test solution and filling the vessel immediately with fresh standard test solution at room temperature.

11.5 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 document, e.g. "Tested in accordance with ISO 28706-3:2017, Clause 11";

c) a description of the test solution;

d) the temperature at which the test was performed, in °C;

e) the duration of the test, in hours;

f) the results, giving the loss in mass per unit area as calculated for the time period over which testing was conducted (see Clause 9), in grams per square metre, for each individual determination, plus the arithmetic mean, rounded to the nearest 0,1 g/m^2;

g) any deviations from the procedure specified;

h) any unusual features observed during the test;

i) the date of the test.

Bibliography

[1] ISO 28706-1:2008, *Vitreous and porcelain enamels — Determination of resistance to chemical corrosion — Part 1: Determination of resistance to chemical corrosion by acids at room temperature*

[2] ISO 28706-2:2017, *Vitreous and porcelain enamels — Determination of resistance to chemical corrosion — Part 2: Determination of resistance to chemical corrosion by boiling acids, boiling neutral liquids, alkaline liquids and/or their vapours*

[3] ISO 28706-4:2016, *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*

[4] ISO 28706-5:2010, *Vitreous and porcelain enamels — Determination of resistance to chemical corrosion — Part 5: Determination of resistance to chemical corrosion in closed systems*

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