

# 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**

The European Standard EN 14483-3:2004 has the status of a  
British Standard

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## National foreword

This British Standard is the official English language version of EN 14483-3:2004. It supersedes BS 1344-5:1984 and BS EN ISO 4535:2000 which are withdrawn.

The UK participation in its preparation was entrusted to Technical Committee STI/36, Vitreous enamel coatings, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

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### Summary of pages

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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

Emaux 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

Emails und Emailierungen - Bestimmung der Beständigkeit gegen chemische Korrosion - Teil 3: Bestimmung der Beständigkeit gegen chemische Korrosion durch alkalische Flüssigkeiten unter Verwendung eines Gerätes mit hexagonalem Gefäß

This European Standard was approved by CEN on 1 April 2004.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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## Foreword

This document (EN 14483-3:2004) has been prepared by Technical Committee CEN/TC 262 "Metallic and other inorganic coatings", the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 2004, and conflicting national standards shall be withdrawn at the latest by December 2004.

This European Standard is divided into the following five parts, in accordance with the different apparatus and the different physical test conditions (temperature, pressure, stirring) that are used:

EN 14483 *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, 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*

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## EN 14483-3:2004 (E)

### Introduction

Corrosion of vitreous and porcelain enamel by aqueous solutions is a dissolution process. The main component of the vitreous and porcelain enamel,  $\text{SiO}_2$ , forms a three-dimensional silica network. After hydrolysis it decomposes and forms silicic acid or silicates, respectively. 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, respectively. 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 of the vitreous and porcelain enamel proceeds linearly during the corrosion time, for other aqueous solutions, the attack of the vitreous and porcelain enamel proceeds in a logarithmic manner during the corrosion time. Only for the first series of solutions, a scientific exact rate of loss in mass per unit area ( $\text{g/m}^2\cdot\text{h}$ ) can be calculated as well as a corrosion rate ( $\text{mm/a}$ ).

The most important parameters influencing aqueous corrosion of vitreous and porcelain enamel are vitreous and porcelain enamel quality, temperature and pH-value. Besides, inhibition effects resulting from limited solubility of silica can 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 EN 14483-4:2004) the silica network of the vitreous and porcelain 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 testing times. Therefore test results are expressed in terms of a rate of loss in mass per unit area (weight 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 EN 14483-1:2004) or also in stronger acids like sulfuric acid (see clause 10 of EN 14483-1:2004), there is only minor attack on the silica network of the vitreous and porcelain enamel. Other constituents are leached to some extent from the surface. High resistant vitreous and porcelain enamels will show no visual change after exposure. On less resistant vitreous and porcelain enamels some staining or surface roughening will occur.
- c) In boiling aqueous acids (see EN 14483-2) the silica network of the vitreous and porcelain enamel is being attacked, and silica as well as the other vitreous and porcelain enamel components are released into solution. However, 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, corrosion markedly drops.

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

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

Examples for enamel corrosion proceeding in a logarithmic manner c.1) and linearly c.2) are:

- **c.1) Boiling citric acid (see clause 10 of EN 14483-2:2004) and boiling 30 % sulfuric acid (see clause 11 of EN 14483-2:2004):**

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 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.

- **c.2) Boiling 20 % hydrochloric acid (see clause 12 of EN 14483-2:2004):**

Since this is an azeotropic boiling acid, acid concentration in liquid and 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 (weight 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 EN 14483-5), aqueous acid attack is severe. To avoid inhibition testing time is restricted to 24 h and the ratio of attacking acid versus attacked vitreous and porcelain enamel surface is chosen comparatively high (similar to a chemical reaction vessel). In addition, only low silica water is taken for the preparation of test solutions. Under these provisions attack will proceed linearly with time of exposure. Therefore, test results, either with 20 % hydrochloric acid (see clause 8 of EN 14483-5:2004), artificial test solutions (see clause 9 of EN 14483-5:2004), or process fluids (see clause 10 of EN 14483-5:2004) 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 EN 14483-2:2004) the silica network is fairly stable. The vitreous and porcelain enamel surface is leached, 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 high resistant vitreous and porcelain enamels. Or, if the vitreous and porcelain enamel in test is weak, leached alkali from the vitreous and porcelain enamel can raise pH-values to alkaline levels increasing the attack by the liquid phase. Both liquid and vapour phase test can give valuable information.
- f) Since the attack can be linear or not, results are only expressed in terms of loss in mass per unit area and the testing time should be indicated.
- g) For the standard detergent solution (see clause 9 of EN 14483-3:2004) it is not certain if the linear part of the corrosion curve is reached during the testing for 24 h or 168 h. Calculation of the corrosion rate is therefore not included in the test report.
- h) For the undefined acids (see clause 14 of EN 14483-2:2004) and undefined alkaline solutions (see clause 10 of EN 14483-3:2004 and clause 10 of EN 14483-4:2004), it also is not known if a linear corrosion will be reached during the testing period. Calculation of the corrosion rate is therefore not included in those test reports.

For vitreous enamels fired at temperatures below 700 °C, the testing parameters (media, temperatures, and times) of this 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 and/or conditions" of the parts 1, 2, 3, or 4 of this standard.

EN 14483 Part 1 to Part 5 has been developed from EN ISO, 4535, EN ISO 8290, ISO 2722, ISO 2733, ISO 2734, ISO 2742, ISO 2743, ISO 2745, ISO 4533 and ISO 13806.

## EN 14483-3:2004 (E)

### 1 Scope

This part of EN 14483 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 in this section is a hexagonal vessel in which 6 enamelled specimens are simultaneous tested.

NOTE 1 The resistance to any alkaline liquid can be tested. However, the test method was original made 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 property and complexing behaviour, can be considered as a typical composition for the detergents at 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 property of the standard test solution is obtained by the addition of alkylsulphonate. 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 considerable 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

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods* (ISO 3696:1987).

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

ISO 2723, *Vitreous and porcelain enamels for sheet steel — Production of specimens for testing*.

ISO 2724, *Vitreous and porcelain enamels for cast iron — Production of specimens for testing*.

ISO 13804, *Vitreous and porcelain enamels for aluminium — Production of specimens for testing*.

### 3 Principle

Six 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.

### 4 Reagents

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

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

**4.2 Grease solvent**, such as ethanol, or water (4.1) containing a few drops of liquid detergent for cleaning the testing apparatus and test specimens.



- 4.3 **Sodium tripolyphosphate** ( $\text{Na}_5\text{P}_3\text{O}_{10}$ );
- 4.4 **Sodium carbonate** ( $\text{Na}_2\text{CO}_3$ ) anhydrous;
- 4.5 **Sodium perborate**, hydrated ( $\text{NaBO}_2 \cdot \text{H}_2\text{O}_2 \cdot 3\text{H}_2\text{O}$ );
- 4.6 **Sodium silicate**, containing about 81 % (m/m)  $\text{Na}_2\text{SiO}_3$ ;
- 4.7 **Alkylsulphonate** ( $\text{CH}_3(\text{CH}_2)_x - \text{C}(\text{SO}_2\text{Na}) \text{H} - (\text{CH}_2)_3 - \text{CH}_3$ ).

## 5 Apparatus and materials

### 5.1 Test apparatus

#### 5.1.1 General description

The apparatus (see Figures 1 to 4) consists of a hexagonal 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. 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.

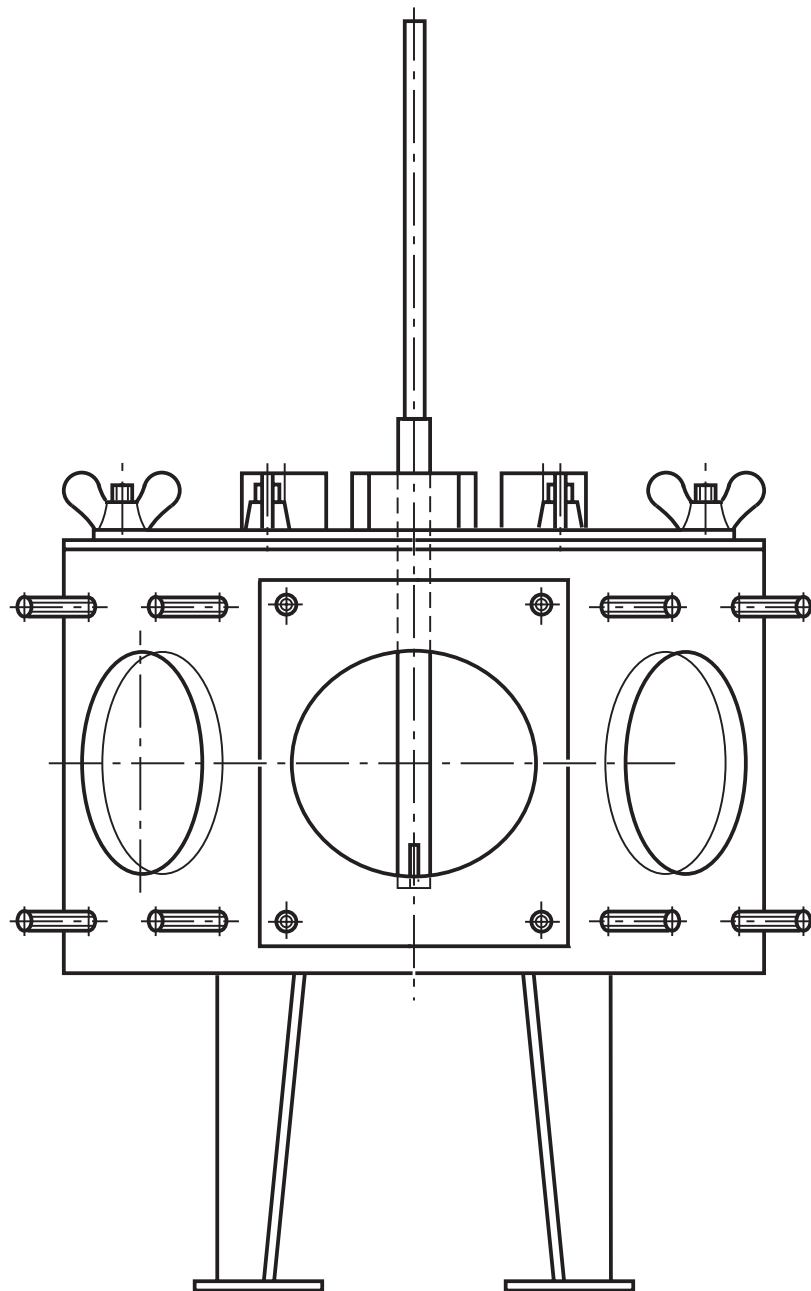


Figure 1 — Hexagonal vessel with lid, stirrer and gripping plate

Dimensions in millimetres

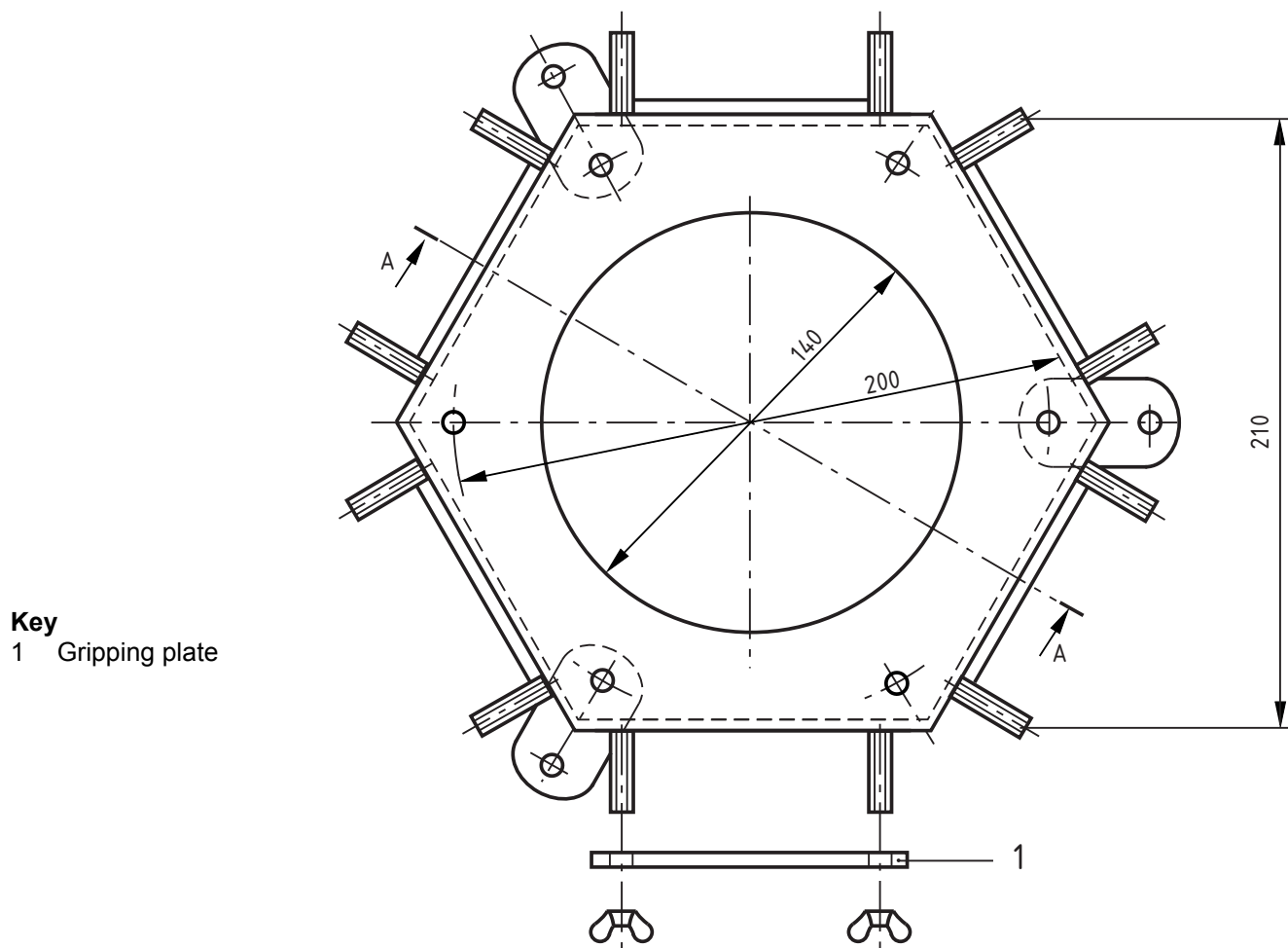


Figure 2 — Top view of hexagonal vessel without lid and paddle stirrer

Dimensions in millimetres

**Key**

- 1 Paddle stirrer
- 2 Socket for temperature controlling device
- 3 Socket for immersion heater
- 4 Lid
- 5 Sealing ring

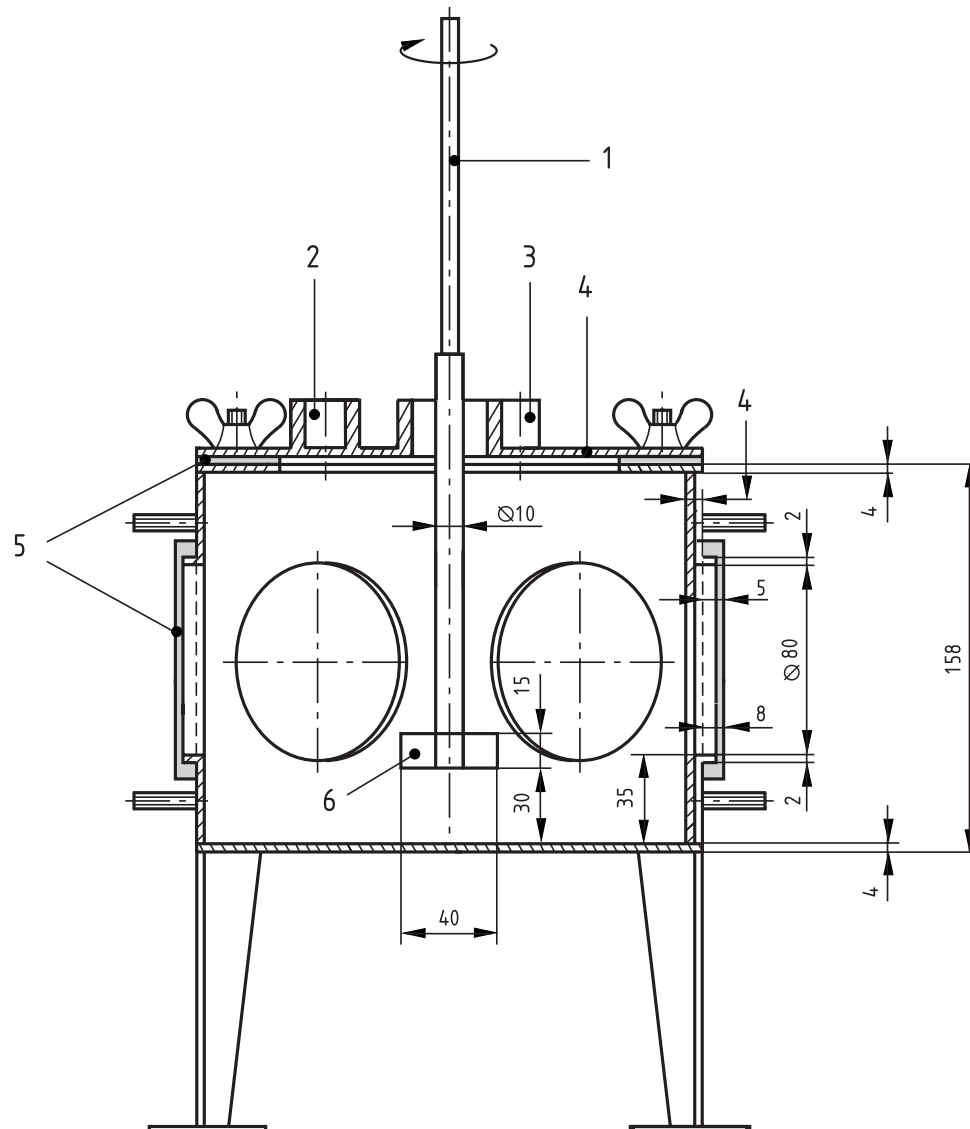


Figure 3 — Section A-A of the hexagonal vessel, lid and paddle stirrer, with additional sealing in the cutting plane

Dimensions in millimetres

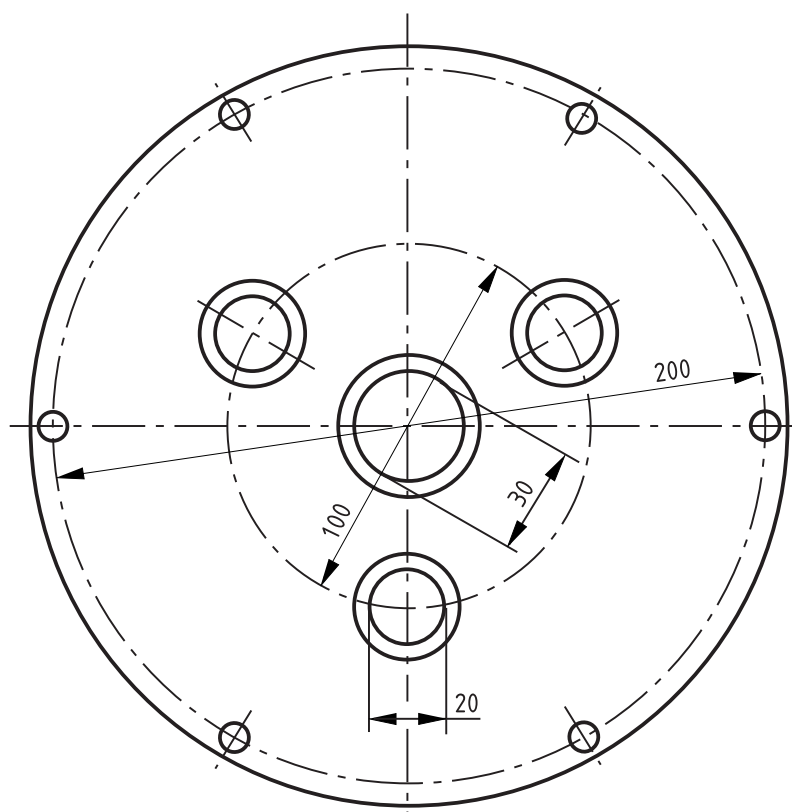


Figure 4 — Top view of lid

The sealing rings (5.1.6) shall be made of a synthetic rubber of hardness 70 IRHD when determined in accordance with ISO 48. The material shall be resistant to alkaline solutions at 100 °C (chloroprene is suitable for example).

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

**5.1.2 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 six threaded bolts welded to the upper surface for fastening the lid. The vessel should preferably have an outlet for drainage.

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

**5.1.4 Gripping plates** (6), of austenitic stainless steel, of thickness 4 mm, and which can be fitted to the sides of the hexagonal vessel.

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

**5.1.6 Sealing rings** (6), 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.

**5.1.7 Paddle stirrer**, of austenitic stainless steel with dimensions shown in Figure 3. It shall operate at a rotational frequency of  $(1350 \pm 50) \text{ min}^{-1}$ .

**5.1.8 Immersion heaters** (2), cylindrical, each of 600 W, made of nickel-plated copper or of austenitic stainless steel.

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**5.1.9 Temperature controlling device**, comprising a contact thermometer with a thermostat accurate to  $\pm 1$  °C. The use of a temperature recording instrument is recommended.

**5.2 Hot air oven**, capable of being maintained at  $(120 \pm 5)$  °C.

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

**5.4 Balance**, accurate to 0,2 mg.

**5.5 Cotton wool**.

## 6 Test specimens

The specimens to be used shall be prepared in accordance with the International Standards for the appropriate basis metal (ISO 2723, ISO 2724 and ISO 13804). Enamel both sides of the test specimens.

## 7 Procedure

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

Before the test, wipe each test specimen with cotton wool (5.5) soaked in the degreasing medium (4.2). Then dry the specimens for 2 h in the hot air oven (5.2), controlled at  $(120 \pm 5)$  °C, cool for at least 2 h in the desiccator (5.3) and weigh to the nearest 0,2 mg. Record the starting mass,  $m_s$ .

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 liquid (see clauses 9 and 10), at room temperature, into the vessel through the inlet in the lid. Heat the test solution to the described temperature (see clauses 9 and 10), stirring continuously, and maintain it at this temperature for the described time (see clauses 9 and 10).

At the end of the required time (see clauses 9 and 10), remove the hot standard test solution and fill the vessel immediately with water (4.1) at room temperature. Stir the water (4.1) 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 (4.1) and then rinse with the degreasing medium (4.2). Dry the specimens for 2 h in the hot air oven (5.2), controlled at  $(120 \pm 5)$  °C, and then leave them in the desiccator (5.3) 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 and calculate the mean value of 3 measurements of the diameter exposed to attack. This mean value shall fall within  $\pm 1$  mm of 80 mm. Calculate the exposed area of attack,  $A$ , using this mean value of the diameter.

## 8 Expression of results

For each test calculate the results as the total loss in mass per unit area,  $\Delta\rho_A$ , in g/m<sup>2</sup>, for the total duration of the test, using 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;

and

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

In order to distinguish between the testing results of different testing periods, the number of testing hours shall be stated as a subscript to the symbol; for example, for 24 h testing time,  $\Delta\rho_{A24}$ .

For the evaluation, the results of the specimens which show defects such as pinholes down to the metal, chipped edges, or edge corrosion, shall be omitted. The corresponding number of new test specimens shall be tested.

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

## 9 Standard detergent solution test

### 9.1 General

Carry out this test following the procedure described in clause 7.

### 9.2 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 % (m/m)  $Na_2SiO_3$ ;
- 4,5 g of alkylsulphonate ( $CH_3(CH_2)_x - C(SO_2Na)H - (CH_2)_3 - CH_3$ ).

The solution shall be made up using water (4.1) and reagents of analytical grade. All quantities refer to a substance with an efficiency of 100 %.

Prepare a fresh test solution for each test.

NOTE Each 24 h of test uses 4,5 l of the test solution.

### 9.3 Test temperature

The test solution shall be heated in the vessel to  $95\text{ }^\circ\text{C}$  and shall be maintained at that temperature during the testing time.

### 9.4 Duration of the test

The controlled heating time at  $95\text{ }^\circ\text{C}$  (i.e. without the duration for heating-up) shall be 24 h.

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

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 10.

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### 9.5 Test report

The test report shall include the following information:

- a) all information necessary for the identification of the vitreous and porcelain enamel sample tested;
- b) a reference to clause 9 of this European Standard, EN 14483-3 (including its year of publication) e.g.  
"Tested in accordance with clause 9 of EN 14483-3:2004 – standard detergent solution";
- c) the duration of the test in hours;
- d) the result(s), including the results of the individual determinations, giving the loss in mass per unit area as calculated for the time period over which testing was conducted (8), in grams per square metre, rounded to the nearest 0,1 g/m<sup>2</sup>, giving the arithmetic mean together with the individual values;
- e) any deviations from the procedure specified;
- f) any unusual features observed during the test;
- g) the date of the test.

## 10 Other test solutions and/or conditions

### 10.1 General

Carry out this test following the procedure described in clause 7

### 10.2 Test solution

Prepare 4,5 l of an agreed alkaline test solution using water (4.1) and reagents of analytical grade. No test solutions may be used that could damage the apparatus.

Prepare a fresh test solution for each test.

NOTE Each 24 h of test uses 4,5 l of the test solution.

### 10.3 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 testing time.

### 10.4 Duration of the test

The controlled 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 that testing period, the result of the test is "<1,6 g/m<sup>2</sup>".

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 testing 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.



## 10.5 Test report

The test report shall include the following information:

- a) all information necessary for the identification of the vitreous and porcelain enamel sample tested;
- b) a reference to clause 10 of this European Standard, EN 14483-3 (including its year of publication) e.g. "Tested in accordance with clause 10 of EN 14483-3:2004 – other test solution";
- 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 result(s), including the results of the individual determinations, giving the loss in mass per unit area as calculated for the time period over which testing was conducted (8), in grams per square metre, rounded to the nearest 0,1 g/m<sup>2</sup>, giving the arithmetic mean together with the individual values;
- g) any deviations from the procedure specified;
- h) any unusual features observed during the test;
- i) the date of the test.

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