BRITISH STANDARD

BS EN
14483-5:2004

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

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

The European Standard EN 14483-5:2004 has the status of a British Standard

ICS 25.220.50

BSI
British Standards

National foreword

This British Standard is the official English language version of EN 14483-5:2004. It supersedes BS ISO 13806:1999 which is withdrawn.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.

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

Emails und Emaillierungen - Bestimmung der Beständigkeit gegen chemische Korrosion - Teil 5: Bestimmung der Beständigkeit gegen chemische Korrosion in geschlossenen Systemen

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

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Contents

Foreword

This document (EN 14483-5:2004) 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of an identical text or by endorsement, at the latest by December 2004, and conflicting national standards shall be withdrawn at the latest by December 2004.

Annex A is informative.

This document includes a Bibliography.

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*

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Introduction

Corrosion of vitreous and porcelain enamel by aqueous solutions is a dissolution process. The main component of the vitreous and porcelain enamel, 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 ($g/m^2.h$) can be calculated as well as a corrosion rate (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.

1 Scope

This part of EN 14483 describes a test method for the determination of resistance to attack in closed systems by acid and neutral liquids, as well as by actual process mixes, the given corrosive agent generally applied at a temperature above its boiling point.

It is also applicable to the determination of resistance to mildly alkaline fluids provided that the material of the test equipment is suitable for such a test (see also 4.1.2).

This European Standard primarily applies to the testing of enamels designed for use in chemical process technology.

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

3 Principle

Enamelled test specimens are exposed to attack by a liquid corrosive at temperatures above the normal boiling point under defined autoclave conditions.

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

4 Apparatus

4.1 Test vessel

4.1.1 Design

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

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

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

4.1.2 Material

The test vessel shall be made of a material resistant to the test solution and not releasing any substances that might influence the corrosion of the enamel. In particular, glass or ceramic flasks and fluorinated plastics for coating

or fitting shall be avoided. As a component of seals, polytetra-fluorethylene (PTFE) is the only suitable fluorinated plastic material for tests with mineral acids, e.g. sulphuric acid and hydrochloric acid.

NOTE Vessels with tantalum fittings or with electrolytically deposited tantalum coatings or vessels made of solid tantalum observe these requirements for acid and neutral solutions over a wide range of applications.

4.1.3 Fittings in the test vessel

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

4.1.4 Heating device

The type of heating device and its power shall be selected such that the test temperature is reached within 1 h and controllable to 1 °C, where the test temperature is defined as the temperature of the test solution at the interface to the enamel surface.

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

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

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

4.4 Desiccator, capable of enclosing the test specimens.

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

5 Test specimens

5.1 Test specimen shape and preparation

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

NOTE The manufacturer of the samples should ensure that the composition of the enamel and the process by which it is applied is the same as on other pieces of production.

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

5.2 Number of test specimens

At least two test specimens shall be tested where the actual number of test specimens depends on the number of individual values required to take the arithmetic mean (see 7.2)

5.3 Conditioning

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

6 Procedure

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

After closing the test vessel, heat to test temperature (see clauses 8 to 10).

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

Switch off the heating at the end of the exposure period and allow the test vessel to cool in air.

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

NOTE It is recommended to ensure that the cleaning procedure does not attack the enamel, e.g. by causing scratches.

Dry the test specimens for 2 h in the oven (4.3) at 110 °C ± 5 °C. Allow them to cool for further 2 h in the desiccator (4.4). Remove and weigh them immediately to the nearest 0,02 mg.

Reject all test specimens which have lost mass for reasons not due to corrosion, e.g. caused by chipping or scratches. Test a corresponding number of new test specimens.

Determine the exposed area of enamel and the loss in mass, Δm, of the test specimens such that the sum of the maximum relative errors of measurement at a rate of loss in mass per unit area, v = 0,0285 g/m^2.h, is not greater than 10 % (see also annex A).

7 Expression of results

7.1 Rate of loss in mass per unit area and corrosion rate

Calculate the rate of loss in mass per unit area, *v*, and the corrosion rate, *w*, using equations (1) and (2):

$$v = |\Delta m| / (A \times t) \quad (1)$$

$$w = 3{,}504\ v \quad (2)$$

where

A is the exposed area of enamel, in square metres (m^2);

Δm is the loss in mass, in grams (g);

t is the exposure time, in hours (h);

v is the rate of loss in mass per unit area, in grams per square metres per hours (g/m^2.h);

w is the corrosion rate, in millimetres per year (mm/a).

NOTE It is assumed that enamel is a homogeneous material with a density of 2,5 g/cm^3 in equation (2).

7.2 Calculation of arithmetic mean

Calculate the arithmetic mean of the rate of loss in mass per unit area, *v*, from the individual values provided that the difference between the maximum and minimum values (range) is not greater than the permitted difference.

The permitted relative range related to the lowest measured value, which depends on the number of individual measured values, shall be in accordance with Table 1.

Table 1 — Permitted relative range as a function of the number of measured values	
Number of measured values	Permitted relative range %
2	30
3	37
4	42

If the permitted relative range for two measured values is less than or equal to 30 %, conclude the test after testing two test specimens.

If the permitted relative range is greater than 30 % but less than or equal to 37 % or 42 %, test one and two further test specimens, respectively. Then calculate the arithmetic mean from all three respectively four individual values; otherwise, repeat the test with new test specimens.

7.3 Corrosion rate

Calculate the arithmetic mean of the corrosion rate, w, from the arithmetic mean of the rate of loss in mass per unit area, v, determined as specified in 7.1 using equation (2).

8 Autoclave test with hydrochloric acid

NOTE It is recommended to carry out this test following the procedure described in clause 6.

8.1 Test solution

Hydrochloric acid, analytical grade, 20% (m/m) solution, density range, 1,097 g/ml to 1,099 g/ml (measured with a hydrometer, specified in ISO 649-1). A fresh solution shall be used for each test.

The solution shall be made using demineralized water, conforming to the requirements of EN ISO 3696, with a concentration of SiO_2 by mass of no more than $0{,}1 \times 10^{-3}$ g/l.

8.2 Test temperature

The test temperature shall be 140 °C ± 1 °C.

8.3 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 8 of this European Standard, including its year of publication; e.g.

"Tested in accordance with clause 8 of EN 14483-5: 2004 – autoclave test with hydrochloric acid ";

c) the number of test specimens;

d) the number of test specimens simultaneously in the test vessel;

e) the result(s), including the results of the individual determinations, giving:

— the rate of loss in mass per unit area, in grams per square metres per hour, individual values and arithmetic mean;

— the corrosion rate as arithmetic mean, in millimetres per year;

f) any deviations from the procedure specified;

g) any unusual features observed during the test;

h) the date of the test.

9 Autoclave artificial solution test

NOTE It is recommended to carry out this test following the procedure described in clause 6.

9.1 Test solution

An agreed test solution shall be made up using demineralised water with a concentration of SiO_2 by mass of no more than $0{,}1x10^{-3}$ g/l and reagents of analytical grade.

9.2 Test temperature

The test temperature shall be agreed in advance and shall be recorded in the test report.

9.3 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 9 of this European Standard, including its year of publication; e.g.

"Tested in accordance with clause 9 of EN 14483-5: 2004 – autoclave test with artificial solution ";

c) a description of the artificial test solution;

d) the test temperature;

e) the number of test specimens;

f) the number of test specimens simultaneously in the test vessel;

g) the result(s), including the results of the individual determinations, giving

— the rate of loss in mass per unit area, in grams per square metres per hours: individual values and arithmetic mean;

— the corrosion rate as arithmetic mean, in millimetres per year

h) any deviations from the procedure specified;

i) any unusual features observed during the test;

j) the date of the test.

10 Autoclave test with process fluids

NOTE It is recommended to carry out this test following the procedure described in clause 6.

10.1 Test solution

The composition of the test solution shall be defined in advance and shall be recorded in the test report.

NOTE When carrying out the test using process fluids, even small quantities of their constituents can inhibit or accelerate the rate of loss in mass. The inhibitory effect of silicon dioxide and materials releasing silicon dioxide, for example, is well-known.

10.2 Test temperature

The test temperature shall be fixed in advance and shall be recorded in the test report.

10.3 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 European Standard, including its year of publication; e.g.

"Tested in accordance with clause 10 of EN 14483-5: 2004 – autoclave test with process fluids ";

c) a description of the process fluid;

d) the test temperature;

e) the number of test specimens;

f) the number of test specimens simultaneously in the test vessel;

g) the result(s), including the results of the individual determinations, giving

- the arithmetic mean rate of loss in mass per unit area, in grams per square metre per hour together with individual values;
- the corrosion rate as arithmetic mean, in millimetres per year;

h) any deviations from the procedure specified;

i) any unusual features observed during the test;

j) the date of the test.

Annex A
(informative)

Explanatory notes

This standard does not specify detailed test equipments but permits the user to design test vessels in such a way that these can meet particular requirements of a specific product (test solution) or of an operational process. Examples of test vessels and test specimens are given in the literature (see Bibliography, [1], [2], [3]).

In order to ensure the reproducibility of results obtained for different test vessels, the maximum permissible error of measurement in the test method should be restricted to less than the likely maximum relative deviation of the measured value from the true value. As a simplification, for the test parameters given in this European Standard, the maximum error of measurement is determined solely by the permissible deviation of the test temperature from the specified value. Other influence quantities such as volume/surface ratio and pressure have a relatively small effect on the result if the limit values specified in this European Standard are maintained (see [1], [2], [3]).

The temperature dependency of the corrosion rate can be described in a simple analytical relationship as established by Arrhenius [1]. This shows that for a maximum temperature deviation of 1 °C from the specified value in the temperature range from 100 °C to 180 °C there is a maximum relative deviation of 10 %. The relative error of measurement decreases in inverse proportion to the test temperature. Therefore the technical important corrosion rate value of 0,1 mm/a representing the resistance limit should be determined to the nearest 0,01 mm/a.

The precision of the test method is defined by the maximum error of measurement resulting from the maximum errors of measurement for the individual values measured (i. e. loss in mass, exposure period and area of enamel exposed). This is calculated directly from the maximum relative errors of measurement according to the law of error propagation:

$$\frac{\Delta v}{v} = \frac{\Delta A}{A} + \frac{\Delta t}{t} \qquad \text{(A.1)}$$

where A, m, t and v are as defined in 7.1.

The maximum relative error of measurement for the exposure period as specified in this European Standard is 0,35 %, thus the total of the maximum errors of measurement from loss in mass and area of enamel exposed should not be greater than 9,65 %.

The maximum error of measurement is 2,1 cm^2 for the area of a cylindrical test rod enamelled on all sides with a specified surface area of 25 cm^2 [2] resulting from the measurement uncertainty for weighing the loss in mass specified in 5.3 and clause 6. If a surface area of only 11 cm^2 is specified [2], the area should be determined to within 0,8 cm^2. Where an approximately flat area, described by a 40 mm diameter circle, is exposed to the test solution, the area should be determined to within 0,9 cm^2, i.e. the diameter should be determined to within 0,7 mm.

A distinction should be made between liquid and vapour phase exposure in the case of enamel corrosion occurring in chemical process engineering. Under described process conditions the maximum corrodibility in the liquid phase can be determined when using the current test techniques and considering the specifications in this European Standard. The corrosion rates thus obtained are representative for enamelled containers and vessels in chemical process engineering with a ratio between volume and exposed area of enamel not greater than 40 cm and for a vessel not greater than 6 m^3.

The experimental determination of the maximum corrodibility in the vapour phase still presents problems, mainly due to the difficulties involved in registering the test temperature and mass of condensate. If the composition of the condensate is known, the maximum corrodibility can be determined approximately by preparing a solution of reagents of analytical grade corresponding to the composition of the condensate and using it in the liquid phase.

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