SLOVENSKI STANDARD
SIST EN 14483-5:2004

01-september-2004

Steklasti in porcelanski emajli ï Ugotavljanje odpornosti proti kemiļ ni koroziji ï 5. del: Ugotavljanje odpornosti proti kemiļ ni koroziji v zaprtih sistemih

Vitreous and porcelain enamels - Determination of resistance to chemical corrosion - Part 5: Determination of resistance to chemical corrosion in closed systems

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

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

Ta slovenski standard je istoveten z: **EN 14483-5:2004**

ICS:

25.220.50 Emajlne prevleke Enamels

SIST EN 14483-5:2004 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14483-5

June 2004

ICS 25.220.50

English version

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of an identical text or by endorsement, at the latest by December 2004, and conflicting national standards shall be withdrawn at the latest by December 2004.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