SLOVENSKI STANDARD
SIST EN 14483-1:2004

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Steklasti in porcelanski emajli - Ugotavljanje odpornosti proti kemiį ni koroziji - 1. del: Ugotavljanje odpornosti proti kemiį ni koroziji s kislinami pri sobni temperaturi

Vitreous and porcelain enamels - Determination of resistance to chemical corrosion - Part 1: Determination of resistance to chemical corrosion by acids at room temperature

Emails und Emaillierungen - Bestimmung der Beständigkeit gegen chemische Korrosion - Teil 1: Bestimmung der Beständigkeit gegen chemische Korrosion durch Säuren bei Raumtemperatur

Emaux vitrifiés - Détermination de la résistance a la corrosion chimique - Partie 1: Détermination de la résistance a la corrosion chimique par les acides a température ambiante

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

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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.

CEN members are the national standards bodies of 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.

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Contents

Foreword

This document (EN 14483-1:2004) 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of an identical text or by endorsement, at the latest by December 2004, and conflicting national standards shall be withdrawn at the latest by December 2004.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*

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 of vitreous and porcelain enamelled articles to attack by an acid at room temperature, and also specifies a method of classifying results.

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 1042 *Laboratory glassware — One-mark volumetric flasks (ISO 1042:1998).*

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

ISO 648 *Laboratory glassware — One mark pipettes.*

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 4788 *Laboratory glassware — Graduated measuring cylinders.*

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

3 Principle

Part of the surface of a test specimen is exposed under defined conditions to attack by an acid solution. Resistance is assessed by methods based on the appearance and cleanability of the enamelled surface.

4 Reagents

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

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

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

4.3 Titanium dioxide, pigment grade.

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

4.5 Sulfuric acid, (H_2SO_4), analytical grade, $c(H_2SO_4)$ = 0,5 mol/l.

5 Materials and apparatus

5.1 Graduated measuring cylinder, capacity 100 ml, conforming to the requirements of ISO 4788.

5.2 One mark volumetric flask, capacity 100 ml, conforming to the requirements of EN ISO 1042.

5.3 **Pipette**, conforming to the requirements of ISO 648.

5.4 **Towel,** of white cotton or flax.

5.5 **Filter paper**, free from fluoride, thickness less than 0,18 mm, approximately 30 mm in diameter (only to be used for testing of curved surfaces).

5.6 **Filter paper**, free from fluoride, thickness greater than 0,38 mm, approximately 25 mm in diameter (only to be used for testing of curved surfaces).

5.7 **Filter paper**, free from fluoride.

5.8 **Pencil**, HB hardness or equivalent.

5.9 **Caps** (e.g. curved glasses), made of polyethylene or other suitable flexible material, or glass, having an external diameter approximately 30 mm.

6 Test specimens

The test specimens shall be commercial items, parts thereof, or test pieces especially prepared in accordance with the appropriate standard for that base material.

The production of test specimens for testing vitreous and porcelain enamels for steel sheet, cast iron and aluminium is specified in ISO 2723, ISO 2724, and ISO 13804 respectively.

Each test specimen shall be cleaned with the grease solvent (4.2), then rinsed with hot water until the water spreads evenly on the surface, and then finally dried by dabbing (not rubbing) with the clean towel (5.4).

7 Procedure

7.1 Attack by the testing solution

Using the pipette (5.3), place a few drops of the attacking medium (see 9.1, 10.1 and 11.1), on each specimen and keep it at a temperature of 23 °C ± 3 °C during the whole period of the test, ensuring that there is a continuous treatment area, the diameter of which shall be less than that of the cap (5.9). Cover the treatment area immediately with the cap.

In the case of curved surfaces, place the thin filter paper (5.5) on the area to be treated. On top of this put the thicker filter paper (5.6). Apply a few drops of the attacking medium (see 9.1, 10.1, 11.1), to the top filter paper (5.6) until both filters are saturated. Cover the filter papers to prevent evaporation, for example with a cap (5.9), and keep the specimen at a temperature of 23 °C ± 3 °C.

After the predefined testing time, remove the cap (5.9) and filter papers (5.5 and 5.6), wash the test specimen with either water (4.1) or tap water, then dry it by dabbing (not rubbing) with filter paper (5.7).

When using tap water, ensure that a residual film is not allowed to form, otherwise the classification may be affected.

7.2 Determination

7.2.1 General

Examine each test specimen within 2 h of the completion of the attack by the testing solution. For the evaluation, consider only that part of the surface which has been subjected to exposure to the attacking medium as a treatment area.