INTERNATIONAL
STANDARD

ISO
13806

First edition
1999-12-01

Vitreous and porcelain enamels — Corrosion tests in closed systems

Émaux vitrifiés — Essais de corrosion en milieux fermés

Reference number
ISO 13806:1999(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 13806 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*.

Annex A of this International Standard is for information only.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet iso@iso.ch

Printed in Switzerland

Vitreous and porcelain enamels — Corrosion tests in closed systems

1 Scope

This International Standard specifies a test method for the determination of the resistance of enamels used in chemical process technology to acid and neutral materials, as well as to actual process mixtures in closed systems at temperatures above their boiling point.

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

This International Standard specifies the basic parameters for corrosion tests to ensure adequate repeatability and reproducibility of results. The corrosion rate is calculated from the loss in mass per unit area per unit time, determined according to this test method.

NOTE See ISO 2743 and ISO 2744 for testing of enamels for resistance to boiling acids and neutral liquids in open systems, and ISO 2745 for resistance to alkalis.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative document indicated below. For undated references, the latest edition of normative document referred to applies. Members of ISO and IEC maintain regiters of currently valid International Standards.

ISO 2743, *Vitreous and porcelain enamels — Determination of resistance to condensing hydrochloric acid.*

ISO 2744, *Vitreous and porcelain enamels — Determination of resistance to boiling water and water vapour.*

ISO 2745, *Vitreous and porcelain enamels — Determination of resistance to hot sodium hydroxide.*

3 Designation

The corrosion tests of enamels in closed systems according to this International Standard are designated as follows:

Test ISO 13806

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 (A) of enamel, in square centimetres, shall be $V/A = 40\ \text{cm} \pm 2\ \text{cm}$. The vessel shall be filled so that, when closed at an ambient temperature of 18 °C to 28 °C, at least 20 % of its volume remains available as a vapour head space. To meet this requirement, the size of the test apparatus shall be selected corresponding to that of one of the test specimens. Several enamelled specimens may be placed simultaneously in the same test vessel.

The test vessel may be a pressure vessel therefore the national safety instructions shall be observed.

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. Polytetra-fluorethylene (PTFE) shall only be used as a component of seals.

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

4.1.3 Fittings

The test vessel may be provided with a protective rod for the temperature probe plus a specimen holder and other fittings, e.g., agitator and 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 is controllable to 1 °C, where the test temperature is defined as the temperature of the test solution at the interface between the enamelled surface and the test solution.

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

4.2 Other required equipment

4.2.1 Analytical balance, with an accuracy of 0,001 g.

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

4.2.3 Desiccator.

4.2.4 Sponge.

5 Test solution

5.1 Artificial test solution

Artificial test solutions shall be made from demineralized water containing less than 0,1 mg/l of silicon dioxide, using reagents of analytical grade.

5.2 Process fluids

When carrying out the test using process fluids, it shall be noted that even small quantities of their constituents can inhibit or accelerate the corrosion rate.

NOTE The inhibitory effect of silicon dioxide and materials releasing silicon dioxide, for example, are well-known.

6 Test specimens

6.1 Specimen shape and preparation

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

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

6.2 Number of specimens

At least two specimens shall be tested, but the actual number of specimens will depend on the number of individual values required to make the arithmetic mean (see 8.2).

6.3 Conditioning

Degrease the specimens, rinse them using demineralized water and then dry them in the oven (4.2.2) for at least 2 h at 110 °C ± 5 °C. Once the specimens are dry, cool them in the desiccator (4.2.3) for at least 2 h and weigh them to the nearest 0,001 g immediately after removal from the desiccator.

7 Procedure

The test procedure consists of the following steps.

a) Pour the test solution (see clause 5) into the test vessel (4.1) so as to completely immerse the surface of the specimens to be exposed;

b) After closing the test vessel, heat to temperature (see 4.1.4) ;

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

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

e) Remove the specimens from the test vessel and wash them with the sponge (4.2.4) and demineralized water. Any reaction products still adhering may be removed using mild, non-abrasive cleaning agents. It shall be ensured that the cleaning agents do not attack the enamel.

f) Dry the specimens for 2 h in the oven (4.2.2) at 110 °C ± 5 °C. After allowing them to cool for 2 h in the desiccator (4.2.3), remove and weigh them immediately to the nearest 0,001 g;

g) Reject specimens that have lost mass for any reason clearly not due to corrosion. Test a corresponding number of new specimens;

h) Determine the exposed area of enamel and the loss in mass, Δm, of the specimens such that the sum of the maximum relative errors of measurement at a corrosion rate, w = 0,1 mm/a is not greater than 10 % (see annex A).

8 Expression of results

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

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

$$v = \frac{|\Delta m|}{A \cdot t} \tag{1}$$

$$w = 3{,}504 v \tag{2}$$

where

v is the rate of loss in mass per unit area, in grams per square metre per hour;

Δm is the loss in mass, in grams;

A is the exposed area of enamel, in square metres;

t is the exposure time in hours;

w is the corrosion rate in millimetres per year.

NOTE A mean density for the enamel of 2,5 g/cm^3 is assumed in equation (2).

8.2 Calculation of the arithmetic mean

8.2.1 Rate of loss in mass per unit area

Calculate the arithmetic mean of the rate of loss in mass per unit area, $\bar{v}$, 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 depends on the number of individual measured values as specified in 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 specimens. If the permitted relative range is greater than 30 %, but less than or equal to 37 % or 42 %, test one and two additional specimens, respectively. Then calculate the arithmetic mean from the three or four individual values, respectively. Otherwise, repeat the test with new specimens.

8.2.2 Corrosion rate

Calculate the arithmetic mean of the corrosion rate, $\bar{w}$, from the arithmetic mean of the rate of loss in mass per unit area, $\bar{v}$, determined as specified in 8.2.1 according to equation (2).

9 Test report

The test report shall include the following information:

a) the type of enamel tested;

b) reference to this International Standard, i.e., determined according to Test ISO 13806;

c) the number of specimens tested;

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

e) the type of test solution used;

f) the test temperature;

g) the rate of loss in mass per unit area, in grams per square metre per hour, including the individual values and the arithmetic mean;

h) the corrosion rate as the arithmetic mean, in millimetres per year.

Annex A
(informative)

Explanatory notes

A.1 Notes on designing the test vessel

The general information given in this International Standard permits the user to design test vessels such that they can meet particular requirements of a specific product (test solution) or of an operational process. Examples of test vessels and specimens are given in the literature (see [1],[2],[3]). The test methods specified in ISO 2743, ISO 2744 and ISO 2745 are widely used for comparative testing of enamel coatings.

A.2 Factors that affect reproducibility

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 International Standard, the maximum error of measurement is determined solely by the permissible deviation of the test temperature from the specified value. Other quantities that affect the reproducibility of the results, such as volume/surface ratio and pressure, have a relatively small effect on the results if the limiting values specified in this International Standard are maintained (see [1],[2],[3]).

A.3 Dependence of the corrosion rate on temperature

The temperature dependency of the corrosion rate can be described by the analytical relationship 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 technically important corrosion rate value of 0,1 mm/a, representing the resistance limit, should be determined to the nearest 0,01 mm/a.

A.4 Precision of the test method

The precision of the test method is defined by the cumulative maximum errors of measurement of the individual values; that is, loss in mass, exposure period and area of enamel exposed. The precision is calculated directly from the maximum relative errors of measurement according to the law of error propagation as follows:

$$\Delta v/v = \Delta m/m + \Delta A/A + \Delta t/t$$

See 8.1 for the meanings of the symbols A, m, t and v.

The maximum relative error of measurement for the exposure period specified in this International Standard is 0,35 %. Thus, the total 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 6.3 and 7f). 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; that is, the diameter should be determined within 0,7 mm.

A.5 Liquid versus vapour phase exposures

A distinction should be made between liquid and vapour phase exposure, in the case of enamel corrosion occurring in chemical process engineering. Under prescribed process conditions, the maximum corrodibility in the liquid phase can be determined when using the current test techniques and considering the specifications given in this International 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 no greater than 40 cm and for a vessel capacity no 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 approximately determined by preparing a solution of reagents of analytical grade corresponding to the composition of the condensate and using it in the liquid phase.