INTERNATIONAL STANDARD

ISO 6370-2

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Vitreous and porcelain enamels — Determination of the resistance to abrasion —

Part 2: Loss in mass after sub-surface abrasion

Émaux vitrifiés — Détermination de la résistance à l'abrasion —

Partie 2: Perte de masse après abrasion de la couche superficielle

Reference number
ISO 6370-2:1991(E)

Foreword

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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 6370-2 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Sub-Committee SC 6, *Vitreous and porcelain enamels*.

ISO 6370 consists of the following parts, under the general title *Vitreous and porcelain enamels — Determination of the resistance to abrasion*:

— *Part 1: Abrasion testing apparatus*

— *Part 2: Loss in mass after sub-surface abrasion*

Annexes A, B and C of this part of ISO 6370 are for information only.

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Introduction

Extensive tests have shown that with the comparative method described in this part of ISO 6370 test results may be obtained which agree within ± 5 %. With comparative expenditure, the absolute method gives deviations of ± 30 % and more. Furthermore, absolute quantities for the amount of wear give little information, because abrasives used in practice differ considerably in their effect on enamelled surfaces. Each abrasion test with a standardized method can only be carried out with the aim of providing a general classification of various vitreous and porcelain enamels in relation to each other. Absolute quantities for the amount of wear are not required.

Numerous tests have shown that the three required test periods of 30 min were sufficient to obtain comparable results. If the vitreous and porcelain enamel coat to be tested is thicker than 0,2 mm, it is not necessary to determine the loss in mass after each 30 min test period, because the abrasion under the conditions described in this part of ISO 6370 is directly proportional to the test duration.

A good use of the test method described is the comparison of enamels for similar products.

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Vitreous and porcelain enamels — Determination of the resistance to abrasion —

Part 2:
Loss in mass after sub-surface abrasion

1 Scope

This part of ISO 6370 specifies a test method for determining the resistance of vitreous and porcelain enamel coats to abrasion by rubbing, grinding or other mechanical effects.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 6370. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 6370 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

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

ISO 6370-1:1991, *Vitreous and porcelain enamels — Determination of the resistance to abrasion — Part 1: Abrasion testing apparatus.*

FEPA-Standard 43-GB-1984,[1] *Coated abrasive grains of fused alumina and silicon carbide.*

3 Principle

Mounting of three similarly enamelled test specimens and three reference glass plates in the testing apparatus. Simultaneous exposure of the separated test specimens and reference glass plates to the abrasion attack of a mixture of fused aluminium oxide grains, steel and water for three periods of 30 min.

Calculation of the relative amount of wear W_r from the mean of the mass losses for the three test specimens and the three reference glass plates.

4 Reagents

For cleaning the test specimens and the reference glass plates, use the following reagents.

4.1 Ethanol (C_2H_5OH), with a volume concentration between 96 % and 98 %.

4.2 Distilled water, or water of equivalent purity (grade 3 water complying with the requirements of ISO 3696).

5 Apparatus and materials

5.1 Abrasion testing apparatus, complying with the requirements of ISO 6370-1.

5.2 Balance, accurate to 0,2 mg.

1) Federation of European Producers of Abrasive Products (FEPA).

5.3 Steel balls.

For each test on a set of three specimens and three reference glass plates, the following are required (see 7.3):

500 g of balls of 4 mm in diameter;

400 g of balls of 3 mm in diameter;

250 g of balls of 2 mm in diameter.

All balls shall consist of the same stainless steel of the type used for bearings and shall be hardened, for example, type of steel 20 complying with the requirements of ISO 683-17[1].

5.4 Pipette, of 25 ml nominal capacity, class B at least, complying with the requirements of ISO 648.

5.5 Abrasive, grains of fused aluminium oxide, of grain size P 80, complying with the FEPA-Standard 43-GB-1984.

5.6 Reference glass plates, square plates, with a side length of 100 mm and thickness 3 mm, consisting of float glass[2]. For each test, a set of three reference glass plates is required. For identification of the float-bath surface of the glass plates, see annex A.

5.7 Drying oven, capable of maintaining temperatures of at least 130 °C.

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

6 Test specimens

6.1 Prepare the test specimens in accordance with the International Standards for the appropriate basis metal.

NOTE 1 The production of the specimens for testing vitreous and porcelain enamels for sheet steel and cast iron is specified in ISO 2723[2] and ISO 2724[3], respectively.

6.2 Rinse each test specimen and reference glass plate with water (4.2) and wipe it thoroughly with ethanol (4.1). Dry the test specimens and the reference glass plates in the drying oven (5.7) for 2 h at 120 °C ± 5 °C. Remove them from the oven and allow them to stand for at least 2 h in the desiccator (5.8) and finally weigh each to the nearest 0,2 mg (initial mass).

7 Procedure

7.1 Carry out one test with each set of at least three test specimens and three reference glass plates.

7.2 Fix the test specimens and the reference glass plates on the oscillating table of the abrasion testing apparatus (5.1) with the aid of the retaining rings, sealing rings and clamping devices, so that the cover coat sides of the test specimens and the float-bath surface (see annex A) of the reference glass plates are facing the interior of the retaining rings (see ISO 6370-1:1991, figure 1).

7.3 Fill each retaining ring with an abrading charge and close it with the stopper. The abrading charge consists of

80 g of steel balls (5.3) of 4 mm in diameter;

60 g of steel balls (5.3) of 3 mm in diameter;

35 g of steel balls (5.3) of 2 mm in diameter;

20 ml ± 0,2 ml of water (4.2);

3 g ± 0,01 g of abrasive (5.5).

The limiting deviations in mass for the balls: mass of each single ball.

7.4 Start the oscillating table of the abrasion testing apparatus for a period of 30 min ± 1 min, corresponding to 9 000 rotations ± 300 rotations. Then remove the specimens and reference glass plates, and thoroughly rinse the test specimens, the reference glass plates, the retaining rings and the sealing rings under running water. Dry the test specimens and reference glass plates in air and replace them on the abrasion testing apparatus with a fresh abrading charge (7.3). The steel balls may be used again after thorough cleaning.

If the thickness of the enamel coat to be tested is less than 0,2 mm, it is recommended to weigh the test specimen before the next test period.

Start the oscillating table for a further period of 30 min and then repeat the whole procedure a third time. If the vitreous and porcelain enamel coat being tested has already disappeared, interrupt the test.

7.5 After three test periods of 30 min, remove the test specimens and the reference glass plates from the abrasion testing apparatus. Rinse them thoroughly under running water and then with water (4.2). Dry the test specimens and the reference glass

2) Float glass is glass made by a process in which a ribbon of hot glass is floated upon a heated liquid of density greater than that of the glass.

plates in the drying oven (5.7) for 2 h at 120 °C ± 5 °C. Then allow them to stand for at least 2 h in the desiccator (5.8) and finally weigh each to the nearest 0,2 mg (final mass).

NOTE 2 A porous surface of the test specimen after abrasion can cause an increase in mass due to the absorption of water. This phenomenon shall be stated in the test report.

8 Expression of results

8.1 Calculate, for each test specimen and reference glass plate, the loss in mass, Δm, in milligrams.

Calculate the relative amount of wear W_r using equation (1):

$$W_r = \frac{\Delta m_{S1} + \Delta m_{S2} + \Delta m_{S3}}{\Delta m_{R1} + \Delta m_{R2} + \Delta m_{R3}} \quad \ldots (1)$$

where

Δm_{S1}, Δm_{S2} and Δm_{S3} are the respective losses in mass of the three test specimens S1, S2 and S3 tested;

Δm_{R1}, Δm_{R2} and Δm_{R3} are the respective losses in mass of the three reference glass plates tested.

8.2 Also calculate the value α for the test specimen tested and the reference glass plates tested using equation (2):

$$\alpha = (\Delta m_1 + \Delta m_2 + \Delta m_3)(\Delta m_1^2 + \Delta m_2^2 + \Delta m_3^2 - \Delta m_1 \Delta m_2 - \Delta m_2 \Delta m_3 - \Delta m_1 \Delta m_3)^{-1/2} \quad \ldots (2)$$

The abrasion test is considered as reliable if, for each test specimen tested,

$$\alpha_S \geqslant 60$$

and, for each reference glass plate tested,

$$\alpha_R \geqslant 60$$

If the values α_S and/or α_R are less than 60, carry out a further test with new test specimens. (See also annex B.)

9 Test report

The test report shall include the following information:

a) a reference to this part of ISO 6370;

b) a description of the test specimens;

c) the relative amount of wear, W_r;

d) in case of interruption, duration of the abrasion test;

e) a statement, if appropriate, that the surface of the test specimen was porous after abrasion.

Annex A
(informative)

Identification of the float-bath surface of the reference glass plates

The float-bath surface of the glass may be identified by one of the three following methods.

A.1 Chemical method

A.1.1 Reagents

A.1.1.1 Etching solution, made up of

10 volumes of concentrated hydrochloric acid;

10 volumes of distilled water;

8 volumes of hydrofluoric acid, 40 % (V/V)

thoroughly mixed.

A.1.1.2 Cacotheline, 0,1 % (V/V) solution in distilled water.

A.1.2 Procedure

Place 2 or 3 drops of the etching solution (A.1.1.1) on the surface, followed by 1 or 2 drops of the cacotheline solution (A.1.1.2).

A.1.3 Expression of results

A.1.3.1 Float-bath surface

In 5 s to 10 s, a purple colouration is observed.

A.1.3.2 Top surface

The solution remains yellow.

A.2 Ultraviolet method

Use a lamp with a ultraviolet filter giving a peak output in the range of wavelengths between 254 nm and 365 nm, arranged as shown in figure A.1.

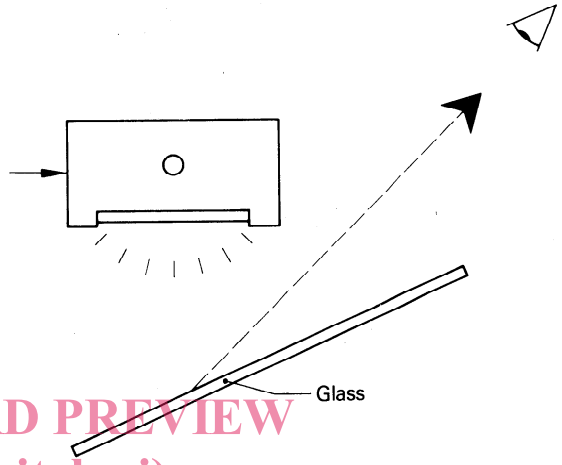

Figure A.1 — Arrangement for the ultraviolet method

When viewed from the angle shown in figure A.1 in a dark room, the float-bath surface exhibits a slight fluorescence.

WARNING — Ultraviolet radiation in this region of the spectrum will damage the eyes and suitable protective ultraviolet-filter goggles must be worn!

A.3 Energy dispersion analysis method

Comparison of the two surfaces of the glass by energy dispersion analysis will easily show up the tin content of the float-bath surface which is not present on the other surface.

Annex B
(informative)

Reasons for the test conditions specified in 8.2

The values α_S and α_R are closely connected with basic terms of the theory of error calculation. To calculate the statistical error of the mean arithmetic values

$$\overline{\Delta m}_S = \frac{1}{3}(\Delta m_{S1} + \Delta m_{S2} + \Delta m_{S3})$$

and

$$\overline{\Delta m}_R = \frac{1}{3}(\Delta m_{R1} + \Delta m_{R2} + \Delta m_{R3})$$

according to the general equation

$$s_{\bar{x}} = \frac{1}{n(n-1)}\left[\sum_{i=1}^{n}(x_1 - \bar{x})^2\right]$$

the following equations are valid:

$$s_{\overline{\Delta m}} = \frac{1}{3}(\Delta m_1^2 + \Delta m_2^2 + \Delta m_3^2 - \Delta m_1\Delta m_2 - \Delta m_2\Delta m_3 - \Delta m_1\Delta m_3)^{1/2}$$

$$s_{\overline{\Delta m}_S} = \overline{\Delta m}_S\left(\frac{1}{\alpha_S}\right)$$

$$s_{\overline{\Delta m}_R} = \overline{\Delta m}_R\left(\frac{1}{\alpha_R}\right)$$

For a confidence level of 95 % of three measurements, the fractile of the Student-Fisher distribution is $t_{95} = 4{,}3$. Consequently, the following inequations are obtained if relative errors of the mean values of less than 7 % are required.

$$\frac{4{,}3\,s_{\overline{\Delta m}_S}}{\Delta m_S} \leqslant 0{,}07$$

$$\frac{4{,}3\,s_{\overline{\Delta m}_R}}{\Delta m_R} \leqslant 0{,}07$$

This leads directly to

$$\frac{4{,}3}{\alpha_S} \leqslant 0{,}07$$

and

$$\frac{4{,}3}{\alpha_R} \leqslant 0{,}07$$

or, respectively

$$\alpha_S \geqslant 60$$

and

$$\alpha_R \geqslant 60$$

According to the law of Gauss for the error propagation and assuming a probability of 90 %, the error for the determination of the relative amount of wear W_r is less than

$$\sqrt{(0{,}07)^2 + (0{,}07)^2} \approx 0{,}1, \text{ or } 10\ \%$$

if the stated requirements for α_S and α_R are fulfilled.