
International Standard



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Vitreous and porcelain enamels — Determination of resistance to hot detergent solutions used for washing textiles

Émaux vitrifiés — Détermination de la résistance aux solutions chaudes de détergent utilisées pour le lavage des textiles

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4533 was developed by Technical Committee ISO/TC 107, *Metallic and other non-organic coatings*, and was circulated to the member bodies in February 1982.

It has been approved by the member bodies of the following countries:

Australia	India	South Africa, Rep. of
Czechoslovakia	Ireland	Spain
Egypt, Arab Rep. of	Italy	Sweden
France	Netherlands	Switzerland
Germany, F.R.	Poland	
Hungary	Romania	

The member bodies of the following countries expressed disapproval of the document on technical grounds:

United Kingdom
USA

Vitreous and porcelain enamels – Determination of resistance to hot detergent solutions used for washing textiles

0 Introduction

The test method described in this International Standard is used for the determination of the resistance of vitreous and porcelain enamels to hot detergent solutions, within the neutral and alkaline range, used for washing textiles.

Since detergents are continually subject to alterations in their composition, a standard test solution is specified which, in respect to its alkalinity, wetting property and complexing behaviour, may be considered as a typical composition for the detergents at present on the market. The pH value and alkalinity of the standard test solution result from the proportions of sodium tripolyphosphate, sodium carbonate and sodium perborate present; sodium tripolyphosphate acts simultaneously as a complexing agent. The wetting property of the standard test solution is obtained by the addition of alkylsulphonate. A higher sodium perborate content is not considered necessary since the effect of oxygen on enamel is unimportant, and an increase in the perborate content does not cause any considerable alteration in the alkalinity of the standard test solution. The testing of different enamels using this standard test solution and other test solutions (including 5 % sodium pyrophosphate solution) has justified the use of this standard test solution for determining the resistance of enamels to hot detergent solutions.

During the development of this test method, the industrially manufactured standard detergent, of the composition specified for the IEC method for measuring the performance of electric washing machines,¹⁾ was examined to determine whether it could be used as the test solution for this test. Full investigation of this problem has shown that the test solution tried out for many years in comparison trials, and specified in this International Standard, should be retained.

Different considerations have necessitated different specifications for the composition of the detergent used for testing the technical washing characteristics of washing machines and that for testing the resistance of enamels to detergent solutions used for washing textiles. The performance test for washing machines is carried out by washing samples of test materials under given conditions, the alteration in these materials due to

the washing process being the test criterion. The composition of the test solution for testing the resistance of enamels to detergent solutions was established chiefly by considering the effect of individual components on the enamel.

For this method of test, the apparatus specified was suggested at the beginning of the development of the method of test. It allows the movement of the washing lye, which is present in all machine washing processes, to be imitated, and allows six test specimens to be tested simultaneously. Efforts over many years to find a simpler and cheaper apparatus have not been successful.

The standard test solution is cold when put into the testing apparatus to correspond to the conditions used in practice, where the washing machine is filled with cold water at the start of the washing process.

Individual conditions for the test method have been based on results from a series of comparative trials which show that it is absolutely essential to observe and maintain the specified test conditions if comparable results are to be obtained. This also applies to the cleaning of test specimens using cotton wool before and after the exposure to the hot detergent solution. If sponges or brushes are used for cleaning, it is not certain that the results will be comparable. Any pieces of cotton wool adhering to the test specimens do not, however, influence the results in any noticeable way.

Test results obtained in accordance with this International Standard are reported as the loss of mass per unit area, in grams per square metre, calculated as the arithmetic mean for duplicate sets, each of six test specimens, after testing for 24 h (1 day), or, if the loss of mass per unit area is low, after testing for 168 h (7 days). Calculation of the rate of corrosion v_K , in grams per square metre per day, could not be included in this International Standard since it is not certain testing for 24 h or for 168 h reaches the linear part of the curve.

As with other test methods, described in ISO 2742,²⁾ ISO 2743,²⁾ ISO 2744,²⁾ and ISO 2745,²⁾ for testing the chemical resistance of enamels, the alteration in gloss or colour

1) IEC Publication 456, *Methods for measuring the performance of electric clothes washing machines for household use.*

2) ISO 2742, *Vitreous and porcelain enamels – Determination of resistance to boiling citric acid.*

ISO 2743, *Vitreous and porcelain enamels – Determination of resistance to boiling 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.*

does not serve as a criterion of the attack by the hot detergent solution. Loss in mass per unit area has been proved, in many comparative trials, to be the only objective criterion. In addition, it has been shown that there is no exact relationship between loss in gloss and the chemical resistance of enamels.

1 Scope and field of application

This International Standard specifies a method for the determination of the resistance of flat surfaces of vitreous and porcelain enamels to hot detergent solutions used for washing textiles.

A standard test solution is specified, but other solutions may be used by agreement between the parties concerned (see clause 0).

2 References

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 4535, *Vitreous and porcelain enamels — Apparatus for determination of resistance to hot detergent solutions used for washing textiles.*

3 Principle

Simultaneous exposure of six similarly enamelled specimens to attack by a detergent solution of specified composition at 95 °C for 24 h (1 day) or for 168 h (7 days), the solution being continuously stirred during the test.

Determination of the loss in mass and calculation of the loss in mass per unit area.

NOTE — The lower the loss in mass per unit area, the higher is the resistance of the vitreous and porcelain enamel to the hot detergent solution.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Standard test solution.

Proceeding at room temperature, prepare 4,5 litres of a solution containing

27,0 g of sodium tripolyphosphate ($\text{Na}_5\text{P}_3\text{O}_{10}$);

9,0 g of anhydrous sodium carbonate (Na_2CO_3);

2,7 g of hydrated sodium perborate ($\text{NaBO}_2 \cdot \text{H}_2\text{O}_2 \cdot 3\text{H}_2\text{O}$);

1,8 g of sodium silicate, containing about 81 % (*m/m*) Na_2SiO_3 ;

4,5 g of alkylsulphonate
 $[\text{CH}_3(\text{CH}_2)_x - \text{C}(\text{SO}_3\text{Na})\text{H} - (\text{CH}_2)_y - \text{CH}_3]$

NOTES

- 1 All quantities refer to a substance with an efficiency of 100 %.
- 2 It is necessary to use 4,5 litres of the test solution for each 24 h of test.
- 3 Fresh test solution should be prepared for each test.

4.2 Ethanol, 96 % (V/V), for cleaning the specimens.

5 Apparatus and material

Ordinary laboratory equipment, together with the apparatus specified in ISO 4535, and

5.1 Hot air oven, capable of being maintained at $120 \pm 5^\circ\text{C}$.

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

5.3 Balance, accurate to 0,2 mg.

5.4 Cotton wool.

6 Test specimens

6.1 The specimens to be used shall be prepared in accordance with the International Standards for the appropriate basis metal. Specimens not enamelled on both sides should be used only for the short test period (24 h).

NOTE — Specimens for testing vitreous and porcelain enamels

- for sheet steel, see ISO 2723;
- for cast iron, see ISO 2724.

6.2 For each determination, two tests with six similarly enamelled specimens shall be carried out.

6.3 Before the test, wipe each test specimen with cotton wool (5.4) soaked in ethanol (4.2). Then dry the specimens for 2 h in the hot air oven (5.1), controlled at $120 \pm 5^\circ\text{C}$, cool for at least 2 h in the desiccator (5.2) and weigh to the nearest 0,2 mg.

7 Procedure

7.1 Press the specimens against the side openings of the hexagonal vessel and secure them by means of the gripping plates so that the vessel is watertight. Pour the standard test solution (4.1), at room temperature, into the vessel through the inlet in the lid. Heat the test solution to 95 ± 1 °C, stirring continuously, and maintain it at this temperature for 24 h.

7.2 After 24 h, remove the hot standard test solution and fill the vessel immediately with water at room temperature. Stir the water for 2 min and then remove it.

Remove the specimens from the vessel and rinse the vessel thoroughly once more. If the vessel is not to be used immediately afterwards, dry it.

7.3 Wipe both sides of the specimens with cotton wool soaked in water and then rinse with the ethanol (4.2). Dry the specimens for 2 h in the hot air oven (5.1), controlled at 120 ± 5 °C, and then leave them in the desiccator (5.4) for 2 h. Weigh each specimen to the nearest 0,2 mg.

7.4 If the average loss in mass per unit area is less than 5 mg after 24 h, repeat the test with new sets of specimens, increasing the test period to 168 h. Replace the standard test solution after each 24 h period, by removing the hot standard test solution and filling the vessel immediately with fresh standard test solution at room temperature.

8 Expression of results

8.1 Calculation

The area exposed to the attack of the test solution is assumed to be 50 cm². The loss of mass per unit area, for a 24 h test ($\Delta\varrho_{A\ 24}$), or for a 168 h test ($\Delta\varrho_{A\ 168}$), expressed in grams per square metre, is given by the formula

$$\frac{m_1 - m_2}{5}$$

where

m_1 is the mass, in milligrams, of the specimen before the test;

m_2 is the mass, in milligrams, of the specimen after the test.

If, after the test, specimens show defects such as pinholes through to the metal, chipped edges or edge corrosion, the results for such specimens shall not be used for calculation of the arithmetic mean, and the corresponding number of new specimens shall be tested.

Take as the result the arithmetic mean of the values obtained for the 12 test specimens. Round the result to the nearest 0,1 g/m².

8.2 Precision

Each determination consists of two parallel tests and gives 12 single values which are to be averaged. Nine of the individual values shall be separated by less than 20 %; the 20 % are calculated from the arithmetic mean of the individual values. If not, a further test shall be carried out, the results of which shall be taken into account in calculating a new arithmetic mean.

9 Test report

The test report shall contain the following information:

- a) a reference to this International Standard;
- b) the type and identification of the vitreous or porcelain enamel tested;
- c) the test period, in hours;
- d) the arithmetic mean loss in mass per unit area ($\Delta\varrho_{A\ 24}$ or $\Delta\varrho_{A\ 168}$), in grams per square metre, rounded to the nearest 0,1 g/m², together with the individual values;
- e) details of the test solution used if this was not the standard test solution.

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