
**Vitreous and porcelain enamels —
Determination of resistance to hot sodium
hydroxide**

*Émaux vitrifiés — Détermination de la résistance à la soude caustique
chaude*

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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 2745 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 6, *Vitreous and porcelain enamels*.

This second edition cancels and replaces the first edition (ISO 2745:1973), which has been technically revised.

Annex A of this International Standard is for information only.

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Vitreous and porcelain enamels — Determination of resistance to hot sodium hydroxide

1 Scope

This International Standard specifies a method of test for determining the resistance of flat surfaces of vitreous and porcelain enamels to a hot solution of sodium hydroxide.

NOTE — This method of test may also be used for determining the chemical resistance of enamels to other alkaline agents and can also be carried out at temperatures below 80° C, but this should be stated in the test report.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard 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 1042 : —¹⁾, *Laboratory glassware — One-mark volumetric flasks.*

ISO 2723 : 1995, *Vitreous and porcelain enamels for sheet steel — Production of specimens for testing.*

ISO 2724 : 1973, *Vitreous and porcelain enamels for cast iron — Production of specimens for testing.*

ISO 2734 : 1997, *Vitreous and porcelain enamels — Apparatus for testing with alkaline liquids.*

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

¹⁾ To be published. (Revision of ISO 1042:1983).

3 Principle

Enamelled test specimens are exposed to attack by a sodium hydroxide solution, $c(\text{NaOH}) = 0,1 \text{ mol/l}$, at 80°C for 24 h (1 d).

The loss in mass is determined and used to calculate the rate of loss in mass per unit area.

NOTE — The lower rate of loss in mass per unit area, the higher the resistance of the vitreous and porcelain enamel to hot sodium hydroxide.

4 Reagents

Use only reagents of recognized analytical grade and only distilled water, or water of equivalent purity (grade 3 water complying with ISO 3696).

4.1 Sodium hydroxide solution, $c(\text{NaOH}) = 0,1 \text{ mol/l}$.

Dissolve 4 g of NaOH in distilled water and make up to 1 l.

To prevent this test solution from absorbing carbon dioxide, ensure that it is kept in a sealed container.

Use a fresh aliquot of solution for each test.

NOTE — For preparing the test solution it is advisable to use a standard phial (USA: vial) with 4 g of NaOH. Place the standard phial on a one-mark volumetric flask (see 5.7) and tap the upper and lower membranes of the standard phial with a blunt glass rod to allow the standard solution to fall into the flask. Rinse the glass rod and the standard phial into the flask with carbon dioxide-free distilled water and make the solution up to 1 l.

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up to 1 l.
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4.2 Acetic acid solution, concentration 5 % (V/V), for cleaning the test specimens.

4.3 **Cleaning medium**, for example ethanol, ($\text{C}_2\text{H}_5\text{OH}$), or water containing a few drops of liquid detergent, for cleaning and degreasing the test specimens.

5 Apparatus

5.1 Test apparatus, complying with ISO 2734.

5.2 **Thermostatically controlled liquid bath** (containing demineralized or distilled water), incorporating a stirrer or other rotating device for use with one or more pieces of test apparatus. It shall be capable of being sealed against loss by evaporation and of allowing temperatures up to 100°C to be kept constant to $0,1^\circ \text{C}$.

5.3 **Thermometer**, calibrated and graduated in $0,1^\circ \text{C}$ steps, for use with the thermostatically controlled liquid bath.

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

5.5 **Desiccator**, internal diameter of at least 200 mm.

5.6 **Polypropylene bottle**, of 1 000 ml capacity and capable of being closed.

5.7 One-mark volumetric flask, 1 000 ml capacity and complying with class A of ISO 1042.

5.8 Funnel, of maximum diameter 70 mm.

5.9 Balance, accurate to 0,2 mg.

5.10 Sponge, soft.

6 Test specimens

6.1 Prepare at least two test specimens in accordance with the International Standards for the appropriate base metal, i.e. use circular test specimens in accordance with ISO 2723 and ISO 2724, for sheet steel and cast iron, respectively.

6.2 Rinse the test specimens with water. If necessary, use a cleaning and degreasing medium (4.3). Then dry the test specimens for 2 h in the drying oven (5.4) at $110^{\circ}\text{C} \pm 5^{\circ}\text{C}$. Allow the test specimens to stand for at least 2 h in the desiccator (5.5) and finally, weigh the test specimens to the nearest 0,2 mg (starting mass).

7 Procedure

7.1 Carry out one determination for each test specimen.

7.2 Place the test specimen in the protective envelope of the test apparatus (5.1) such that the side with the cover coat enamel faces the opening.

Fix the test specimen in the test apparatus (5.1) such that the side with unprotected cover coat faces the interior of the apparatus.

Screw down the six wing-nuts evenly to ensure that the test apparatus is watertight.

NOTE — Damage to the enamel on weak or distorted test specimens in the apparatus can be avoided by placing a rubber ring between the protective envelope and the flange plate of the apparatus. 2 mm to 3 mm thick rubber rings made of heat-resistant rubber are suitable for this purpose (inside diameter 80 mm, outside diameter 100 mm, Shore hardness A/70/1 according to ISO 868).

7.3 Place the sealed test apparatus in the thermostatically controlled liquid bath (5.2), heated to $80^{\circ}\text{C} \pm 1^{\circ}\text{C}$, such that the filling nozzle juts out over the water bath by approximately 10 mm. Leave the test apparatus in this position for at least 10 min until it is filled with the test solution (see 7.4).

NOTE — The test apparatus may also be placed in the cold thermostatically controlled liquid bath and heated to $80^{\circ}\text{C} \pm 1^{\circ}\text{C}$.

7.4 Heat about 1 000 ml of the test solution (4.1) to $80^{\circ}\text{C} \pm 1^{\circ}\text{C}$ in the polypropylene bottle (5.6) and then pour it through the funnel (5.8) into the test apparatus which is still in the thermostatically controlled liquid bath. Seal the cylinder of the test apparatus (5.1) with the stopper and cover the opening of the liquid bath.

With its bulb close to the test apparatus in the liquid bath at half the height of the test apparatus, use the thermometer (5.3) to check that the test temperature is maintained at $80^{\circ}\text{C} \pm 1^{\circ}\text{C}$ throughout the test. If two or more pieces of test apparatus are used, place the thermometer between them.

7.5 24 h (1 d) after pouring the test solution into the test apparatus, remove the test apparatus from the bath using hooks; pour away the test solution and rinse the interior of the test apparatus with distilled or demineralized water.

Take the test specimen out of the protective envelope and wipe it three times with the sponge (5.10) previously soaked in cold acetic acid solution (4.2), then rinse it with cold water.

Carefully remove any residues of the protective envelopes from the test specimen, then dry the latter for 2 h in the drying oven (5.4) at $110^{\circ}\text{C} \pm 5^{\circ}\text{C}$. After a further 2 h in the desiccator (5.5) weigh the test specimen to the nearest 0,2 mg (final mass).

7.6 Disregard test specimens which show defects such as pinholes down to the metal, chipped edges or edge corrosion and test a corresponding number of new specimens.

8 Expression of results

8.1 Calculate the rate of loss in mass per unit area for a test time of 24 h (1 d), v_{24} , expressed in grams per square metre per hour, using the following equation:

$$v_{24} = \frac{\Delta m \times 10^4}{50 \times 24} = 8,33 \Delta m \quad \dots (1)$$

where

Δm is the loss in mass of the test specimen (starting mass minus final mass), in grams;

50 is the exposed area of the test specimen, in square centimetres;

24 is the exposure time of the test specimen to the sodium hydroxide, in hours.

8.2 Calculate the arithmetic mean of the two individual values. If the difference between the individual values of the rate of loss in mass per unit area, v_{24} , exceeds 30 % of the arithmetic mean, conduct a further test. Calculate a new arithmetic mean.

9 Test report

The test report shall include the following information:

- reference to this International Standard, i. e. "determined in accordance with ISO 2745 : 1998";
- identification of the enamel tested;
- the rate of loss in mass per unit area, v_{24} , expressed in grams per square metre per hour, rounded to the nearest $1 \times 10^{-3} \text{ g} \cdot \text{m}^{-2} \cdot \text{h}^{-1}$, the arithmetic mean and the number of single values;
- any deviation from the test procedure specified (see note in clause 1).

Annex A
(informative)

Bibliography

- [1] ISO 868 : 1985, *Plastics and ebonite — Determination of indentation hardness of plastics by means of a durometer (Shore hardness)*.
- [2] LORENTZ, R., Korrosion von Chemieemail in alkalischen Lösungen [Corrosion of chemical service glass-enamel in alkaline solutions]. *Werkstoffe und Korrosion*, 1986 (vol. 37), pp. 567 - 578.

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