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

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FOREWORD

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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 2745 was drawn up by Technical Committee ISO/TC 107, *Metallic and other non-organic coatings*, and circulated to the Member Bodies in June 1972.

It has been approved by the Member Bodies of the following countries :

Australia	Israel	Romania
Egypt, Arab Rep. of	Italy	South Africa, Rep. of
France	Japan	Sweden
Germany	Netherlands	Switzerland
Hungary	New Zealand	Turkey
India	Poland	United Kingdom
Ireland	Portugal	U.S.S.R.

No Member Body expressed disapproval of the document.

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

1 SCOPE AND FIELD OF APPLICATION

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 the determination of the chemical resistance of enamels to other alkaline agents and may also be carried out at temperatures below 80 °C, but this should be stated in the test report.

2 REFERENCES

ISO/R 868, *Plastics — Determination of indentation hardness of plastics by means of a durometer (Shore hardness).*

ISO/R 1042, *One-mark volumetric flasks.*

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

3 PRINCIPLE

Two similarly enamelled specimens are simultaneously exposed to attack by a solution of normal sodium hydroxide at 80 °C for 48 h (2 days).

The loss in mass is determined and the corrosion speed calculated therefrom.

The lower the corrosion speed the higher is the resistance of the vitreous and porcelain enamel to hot sodium hydroxide.

4 REAGENTS

4.1 Sodium hydroxide, 1 N solution.

Dissolve 40,00 g of NaOH in distilled water and make up to 1 l.

In order to prevent this test solution from absorbing any carbon dioxide it has to be kept sealed.

A fresh solution is required for each test.

NOTE — For preparing the test solution it is advisable to use a standard phial with 40,00 g of NaOH. Place the phial on a one-mark volumetric flask and knock the upper and lower membranes of the phial with a blunt glass rod to allow the standard solution to fall into the flask. Rinse the glass rod and phial into the flask with carbon dioxide-free distilled water and make the solution up to 1 l. Shake the flask, and titrate the solution with standard hydrochloric acid against methyl orange.

4.2 Acetic acid, 5 % (*m/m*) solution, for cleaning the specimens.

4.3 Distilled or **demineralized water,** for cleaning the specimens.

4.4 Grease solvent, such as trichloroethylene or acetone, suitable for cleaning the specimens when necessary.

5 APPARATUS

5.1 Testing apparatus in accordance with ISO 2734.

5.2 Thermostatically controlled liquid bath for use with one or more testing apparatus, with stirrer or other rotating device, capable of being sealed against loss on evaporation and allowing the temperature to be kept constant up to 100 ± 0,1 °C.

NOTE — In order to prevent corrosion it is recommended that an approximately 1 % (*m/m*) solution of sodium nitrite ($NaNO_2$) be used as the liquid in the bath.

5.3 Thermometer, calibrated and graduated in 0,1 °C, for the thermostatically controlled liquid bath.

5.4 Hot-air oven capable of maintaining a temperature of at least 130 °C.

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

5.6 Beakers.

5.7 One-mark volumetric flask, 1 000 ml capacity, class A, in accordance with ISO/R 1042.

5.8 Funnel with a maximum diameter of 70 mm.

5.9 Balance, accurate to ± 0,2 mg.

5.10 Sponge, soft.

6 TEST SPECIMENS

6.1 The specimens to be used shall be prepared in accordance with the International Standards for the appropriate basis metal.

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 two specimens shall be carried out.

6.3 Each specimen shall be rinsed with distilled or demineralized water. If necessary a suitable grease solvent shall be used. The specimen shall be dried for 2 h in the hot-air oven (5.4) at 110 ± 5 °C, then cooled for at least 2 h in the desiccator (5.5) and weighed to the nearest 0,2 mg (starting mass).

7 PROCEDURE

7.1 Place the specimens each in a protective envelope (5.1) in such a way that the sides with the cover coat enamel are facing the opening.

Fix the specimens in the testing apparatus (5.1) so that the unprotected cover coat sides of the specimen are facing the interior of the cylinder.

Screw down the four wing nuts evenly to make the testing apparatus watertight.

NOTE — Tearing of the enamel in the frame on weak, distorted specimens can be avoided by placing a rubber ring between the protective envelope and the frame plate. 2 to 3 mm thick 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/R 868).

7.2 Place the sealed testing apparatus in the thermostatically controlled liquid bath (5.2), heated to 80 ± 1 °C, in such a way that the frame plates are just submerged. Leave the testing apparatus there for at least 10 min until filling with the test solution (see 7.3).

The testing apparatus can also be placed into the cold thermostatically controlled liquid bath and heated to 80 ± 1 °C.

7.3 Heat about 320 ml of test solution (4.1) to 80 ± 1 °C in a beaker (5.6) and then pour it through the funnel (5.8) into the testing apparatus, which is still in the thermostatically controlled liquid bath, until it reaches the filler. Then seal the cylinder again with the stopper and cover the liquid bath opening.

20 min, at the most, after pouring the test solution into the testing apparatus, the liquid in the bath shall have reached the test temperature of 80 ± 1 °C.

The maintenance of this temperature throughout the test shall be verified using the thermometer (5.3), the bulb of which is located in the liquid bath close to the testing apparatus and at half the height of the latter. If two or more testing apparatus are used, the thermometer shall be placed between them.

7.4 After 48 h (2 days) take the testing apparatus out of the bath using tongs or a hook; pour away the test solution and rinse the cylinder with distilled or demineralized water.

Take the specimens out of the protective envelopes and wipe them three times with the sponge (5.10) and cold acetic acid solution (4.2), then rinse them with cold distilled or demineralized water.

Carefully remove the residues of the protective envelopes from the specimens, then dry the latter for 2 h in the hot air oven (5.4) at 110 ± 5 °C.

After a further 2 h in the desiccator (5.5) weigh the specimens to the nearest 0,2 mg (final mass).

8 EXPRESSION OF RESULTS

8.1 The area exposed to the attack of hot sodium hydroxide is assumed to be 50 cm². If the loss in mass Δm (starting mass − final mass) is stated in milligrams, for a testing time of 48 h (2 days) the corrosion speed $v_{K(2)}$ is calculated in grams per square metre per day according to the following equation :

$$v_{K(2)} = \frac{\Delta m}{10} = 0{,}1\ \Delta m$$

8.2 The two tests give four single values which shall be averaged.

The difference between the minimum and maximum individual values of the corrosion speed shall be less than 30 %; the 30 % 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.

For the evaluation, the results of the specimens which show defects such as pinholes down to the metal, chipped edges or edge corrosion, are omitted. The corresponding number of new specimens shall be tested.

9 TEST REPORT

The test report shall include the following particulars :

- corrosion speed $v_{K(2)}$ in grams per square metre per day, rounded to the nearest 0,01 g/(m²·d), giving the arithmetic mean and the number of single values.

ANNEX

Characteristic of the test method is an extraordinarily good repeatability and reproducibility of the test results obtained.

Although the test solution and the test temperature are an essential part of this International Standard, it is possible in particular cases to run the test with an alkaline attacking agent other than 1 N sodium hydroxide and at temperatures below 80 °C (see note in clause 1). Deviations from the procedure given in this International Standard, however, should be stated in the test report.

Testing the alkaline resistance of enamels involves more difficulties than testing the resistance to acids or water. This fact is mainly due to differences in the formation of deposits of salts on the surface of the specimen which are antisoluble and retard the attack in an uncontrollable way, and to the very strong dependence of the alkaline corrosion on temperature.

Testing at temperatures above 80 °C impairs the reproducibility of the results due to deposits of salts on the enamel surface which are antisoluble. Test temperatures below 80 °C impair the reproducibility because of the slight attack.

A test with 1 N sodium hydroxide solution at 80 °C over 48 h results in sufficiently high corrosion for all enamels in question. A deviation of 10 °C as shown in the following figure results in doubling or halving the corrosion speed. Thus, a deviation of only 1 °C means an error of 10 %. This fact makes it necessary to control the test temperature to 0,1 °C which can only be guaranteed in the agitated thermostatically controlled liquid bath.

The immersion of the whole test equipment in a thermostatically controlled liquid bath makes it necessary to protect the edge of the specimen as well as the back against corrosion with an alkali- and heat-resistant rubber envelope. At the same time the rubber envelope serves as sealing unit towards the test equipment.

The alkaline attack is always more severe in the liquid phase. Therefore, a specimen in the vapour phase could be renounced in favour of two specimens in the liquid phase.

Glass or another enamel in contact with the test solution sometimes impairs the test results considerably. Therefore, it is neither admissible to use a test cylinder of glass or ceramic instead of stainless steel, nor allowed to test two different enamelled specimens together in the same cylinder.

The determination of loss in mass has proved superior to any other method of evaluation, especially after considerably high alkaline attack.

The alkaline corrosion leaves on the enamel surface residues partly acid-soluble, partly more or less removable by washing or by rubbing. Therefore, the procedure given in 7.4 of this International Standard should be followed exactly in order to prevent diversified cleaning procedures from impairing the test result.

Edge chipping is frequently the cause of a considerably high corrosion speed and can be seen with the naked eye on the edge of the specimen and inside the protective rubber envelope.

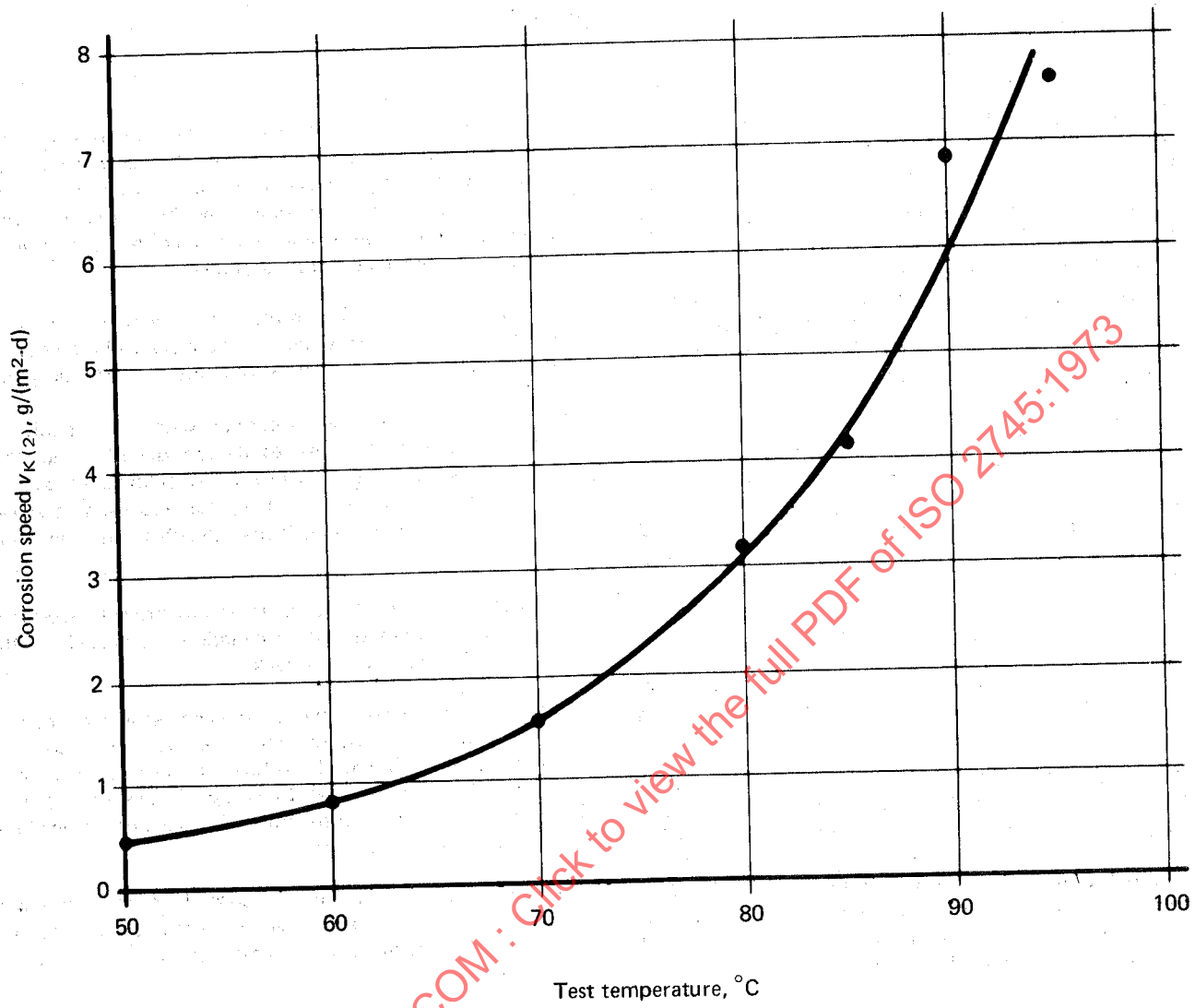

FIGURE – Example concerning the dependence of the alkali-resistance of a tank enamel on the test temperature