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Austenitic stainless steels — Determination of resistance to intergranular corrosion — Part I : Corrosion test in nitric acid medium by measurement of loss in mass (Huey test)

Aciers inoxydables austénitiques — Détermination de la résistance à la corrosion intergranulaire — Partie I : Essai de corrosion en milieu acide nitrique par mesurage de perte de masse (essai de Huey)

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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.

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

Australia	Germany	South Africa, Rep. of
Austria	Hungary	Spain
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No Member Body expressed disapproval of the document.

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Austenitic stainless steels — Determination of resistance to intergranular corrosion — Part I : Corrosion test in nitric acid medium by measurement of loss in mass (Huey test)

1 SCOPE

This International Standard specifies a method for the determination of the resistance to intergranular corrosion of austenitic stainless steels in a nitric acid medium by measurement of the loss in mass (Huey test). It also specifies the purposes which may be assigned to the test.

2 FIELD OF APPLICATION

The method is applicable only to austenitic stainless steels supplied in the form of rolled or forged products and tubes and intended to be used in an oxidizing medium (for example relatively concentrated nitric acid).

NOTES

1 It is important to note that the result of the corrosion test is only strictly valid for the corrosive medium used in the test. It constitutes a basis for estimating the resistance to intergranular corrosion but may not be used to check resistance to other forms of corrosion (general corrosion, by pitting, stress corrosion, etc.). It is necessary for the user to adapt the specified corrosion test to the use which will be made of the metal. This test should, in no case, be considered as an absolute criterion of the quality of the metal if other test methods can also be used. (In some cases the Huey test is the only available test.)

2 In certain particular cases, this test is used to verify that the metallographic structure of the product is well suited to the use.

3 GENERAL

3.1 The term "intergranular corrosion test" denotes the corrosion tests carried out by means of the preferential attacking of the grain boundaries.

Austenitic stainless steels may be subject to such an attack when they are kept at a temperature between about 500 and 800 °C. This heat cycle, which may provoke sensitization to intergranular corrosion, may occur during hot-forming (forging, rolling), as the result of incorrect solution treatment or during a welding operation.

NOTE — In the field of application of this test, the intergranular corrosion may be connected with the presence along the grain boundaries of

- chromium-depleted regions due, in general, to precipitation of chromium carbides;
- intermetallic compounds such as sigma phase.

3.2 The interpretation of the results (for example maximum rate of corrosion) shall form the subject of an agreement between the interested parties.

4 PURPOSE OF THE TEST

This intergranular corrosion test may have either of the purposes given in 4.1 and 4.2. If the order specifies this corrosion test, the purpose of the test shall be stated at the time of ordering.

4.1 Verification of the intrinsic resistance of the metal to intergranular corrosion

This verification applies only to austenitic steels which are specially produced for resistance to intergranular corrosion. The metal is inspected after having undergone a heat treatment for sensitization. (See clause 5.)

4.2 Inspection of the efficiency of the solution treatment

This inspection is only carried out on thin products for which the cooling speed may be made sufficiently rapid. It is only of interest for the steels which are not defined in 4.1. The metal is inspected in the state in which it is delivered to the user, without heat treatment for sensitization.

5 HEAT TREATMENT FOR SENSITIZATION

In order to verify the intrinsic resistance to intergranular corrosion (see 4.1), it is necessary to carry out a heat treatment for sensitization for stabilized steels and steels with a very low carbon content. This sensitization treatment is usually obtained by maintaining the test piece for 30 min at a temperature of 700 ± 10 °C followed by rapid cooling (in water). The duration of the rise in temperature shall not exceed 10 min.

Other sensitization treatments, for example for the preparation of weld test pieces, may be provided for by agreement between the interested parties.

6 CORROSION TEST

6.1 Principle

A test piece, prepared as specified in 6.4.2, is weighed, then immersed in a boiling solution of nitric acid for 5 periods each of 48 h. The criterion for evaluating the test is loss in mass determined by weighing after each period.

6.2 Corrosive solution

The corrosive solution is an aqueous solution of 65 ± 0,2 % (*m/m*) nitric acid (ρ_{20} 1,390 5 to 1,392 4 g/ml).

The products used are known as "analytical quality reagents" and shall have the following residual contents :

Fixed residue	⩽ 50 mg/kg
Pb	⩽ 5 mg/kg
Fe	⩽ 2 mg/kg
Mn	negative test
As	⩽ 0,05 mg/kg
Cl^-	⩽ 1 mg/kg
SO_4^{2-}	⩽ 10 mg/kg
PO_4^{3-}	⩽ 2 mg/kg
F^-	⩽ 1 mg/kg

6.3 Apparatus

6.3.1 Conical flask, of capacity at least 1 l, fitted with a "cold finger" immersion condenser.

Other types of condenser, such as a rising condenser with at least 4 balls, may be used. By means of a paper indicator, it shall be checked that no acid fumes are given off during the test. For the comparative measurements, the apparatus shall remain identical for all the tests.

6.3.2 Support for the test piece, generally made of glass.

6.3.3 Heating device, to keep the solution boiling.

6.4 Test piece

6.4.1 *Dimensions*

The test piece taken from the product shall have its largest dimension located in the direction of working. The test piece dimension will be determined as a function of the weighing facilities and the volume of solution to be used. The test piece length shall, however, be at least equal to twice its width, and the total surface area of the sections perpendicular to the direction of working or the fibres shall be less than 15 % of the total surface area of the test piece. In comparative tests, the ratio of the total surface area to the total surface area of the sections shall be kept constant.

6.4.2 *Preparation*

Depending on the purpose of the test (see clause 4), the test piece, either with or without sensitization treatment, shall be prepared as specified in either 6.4.2.1 or 6.4.2.2. Unless stated on the order to the contrary, the method of preparation shall be left to the manufacturer.

6.4.2.1 Mechanical preparation

The test piece shall be descaled mechanically by polishing on all surfaces, including the edges, with grade 120 abrasive paper or cloth.

6.4.2.2 Chemical preparation

The test piece shall be descaled, without any previous mechanical treatment, in a solution of 50 volumes of hydrochloric acid (ρ_{20} 1,19 g/ml), 5 volumes of nitric acid (ρ_{20} 1,40 g/ml) and 50 volumes of water at 50 to 60 °C.

6.4.2.3 Degreasing

The test piece shall then be degreased before being placed in the corrosive solution.

6.5 Procedure

Use a volume of corrosive solution (6.2) of at least 20 ml per square centimetre of surface area of the test piece. In general, only one test piece shall be placed in each conical flask. However, it is possible to treat several test pieces at the same time, on condition that they all come from the same grade of steel and are isolated one from the other at a distance of at least 5 mm.

Determine the mass of the test piece to an accuracy of 0,001 g as well as its surface area to an accuracy of 5 %. Then immerse the test piece in the corrosive solution and bring the solution to the boil. Boil for 5 periods of 48 h each, using a fresh solution for each period. After each period, wash the test piece in water, dry and weigh.

By special agreement between the interested parties, the test may be limited to 3 periods of 48 h.

In the case of several test pieces being tested in the same receptacle, the test shall be completely cancelled if, after a period of time, an exaggerated loss in mass of one of the test pieces is noted. The test shall be repeated completely on the same test pieces which shall be tested separately after having undergone a fresh surface preparation.

6.6 Expression of results

The effect of the attack by the nitric solution is measured by the mean loss in mass per period.

The corrosion rate is given either in millimetres per year (mm a^{-1}) by formula (1) or in grams per square metre per hour (g m^{-2} h^{-1}) by formula (2) :

$$\frac{87\,600\,m}{S \times \rho \times t} \qquad \ldots (1)$$

$$\frac{10\,000\,m}{S \times t} \qquad \ldots (2)$$

where

t is the period of attack, in hours;

S is the surface area of the test piece, in square centimetres;

m is the mean loss in mass per period, in grams;

ρ is the density of the test piece (8 g/cm³).