

INTERNATIONAL STANDARD

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Determination of resistance to intergranular corrosion of stainless steels —

Part 1:

**Austenitic and ferritic-austenitic (duplex)
stainless steels — Corrosion test in nitric acid
medium by measurement of loss in mass
(Huey test)**

*Détermination de la résistance à la corrosion intergranulaire des aciers
inoxydables —*

*Partie 1: Aciers inoxydables austénitiques et austéno-ferritiques (duplex) —
Essai de corrosion en milieu acide nitrique par mesurage de la perte de
masse (essai de Huey)*



Reference number
ISO 3651-1:1998(E)

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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 preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 3651-1 was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 7, *Methods of testing (other than mechanical tests and chemical analysis)*.

This second edition cancels and replaces the first edition (ISO 3651-1:1976), which has been technically revised.

ISO 3651 consists of the following parts, under the general title *Determination of resistance to intergranular corrosion of stainless steels*:

- *Part 1: Austenitic and ferritic-austenitic (duplex) stainless steels — Corrosion test in nitric acid medium by measurement of loss in mass (Huey test)*
- *Part 2: Ferritic, austenitic and ferritic-austenitic (duplex) stainless steels — Corrosion test in media containing sulfuric acid*

Introduction

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

Austenitic and ferritic-austenitic (duplex) stainless steels may be subject to such attack when they are held at a temperature between about 500 °C and 1 000 °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 — Intergranular corrosion in nitric acid may be associated with one or more of the following:

- precipitation of chromium carbides;
- precipitation of intermetallic compounds such as sigma phase, in molybdenum-bearing grades;
- segregation of impurity elements to the grain boundaries.

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

Determination of resistance to intergranular corrosion of stainless steels —

Part 1:

Austenitic and ferritic-austenitic (duplex) stainless steels — Corrosion test in nitric acid medium by measurement of loss in mass (Huey test)

1 Scope

This part of ISO 3651 specifies a method for the determination of the resistance to intergranular corrosion of austenitic and ferritic-austenitic (duplex) stainless steels in a nitric acid medium by measurement of the loss in mass (Huey test). It also specifies the purpose which may be assigned to the test.

The method is applicable only to austenitic and ferritic-austenitic (duplex) stainless steels supplied in the form of rolled or forged products, tubes and cast products and intended for use in a strongly oxidizing medium (for example, relatively concentrated nitric acid). In general the Huey test should not be used for grades containing molybdenum unless the material tested is to be used in nitric acid service.

NOTE — 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, 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 alloy. This test should, in no case, be considered as an absolute criterion of the quality of the alloy.

2 Purpose of the test

This intergranular corrosion test may have either of the purposes given in 2.1 or 2.2. If an order specifies this corrosion test, the purpose of the test shall be stated at the time of ordering.

2.1 Verification of the intrinsic resistance of the alloy to intergranular corrosion

This verification applies only to austenitic steel grades which are specially produced for resistance to intergranular corrosion in strongly oxidizing media. The specimen is inspected after having undergone a heat treatment for sensitization (see clause 3).

2.2 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. The specimen is inspected in the state in which it is delivered to the user, without heat treatment for sensitization.

3 Heat treatment of sensitization

In order to verify the intrinsic resistance to intergranular corrosion (see 2.1), it is necessary to carry out a heat treatment for sensitization for stabilized steels and steels with a very low carbon content ($C \leq 0,03 \%$). This sensitization treatment is usually obtained by maintaining the test piece for 30 min at a temperature of $700\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$ followed by rapid cooling (in water). The duration of the rise in temperature shall not exceed 10 min.

Other sensitization treatments may be used by agreement between the interested parties.

Welded pieces shall not be submitted to a heat treatment of sensitization.

4 Corrosion test

4.1 Principle

A test piece, prepared as specified in 4.2.2, is weighed, then immersed in a boiling solution of nitric acid for 5 periods each of 48 h. The criterion for evaluating the test result is loss in mass determined by weighing after each period.

4.2 Test pieces

4.2.1 Dimensions

The test piece taken from the wrought product shall have its largest dimension located in the direction of forming. For wrought products and castings the test piece shall be cut as near as possible to the surface. The test piece dimension shall 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 rolling 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 area of the sections shall be kept constant.

4.2.2 Preparation

Depending on the purpose of the test (see clause 2), the test piece, either with or without sensitization treatment, shall be prepared as specified in 4.2.2.1 or 4.2.2.2.

Unless otherwise stated on the order, the method of preparation shall be left to the manufacturer and shall be recorded on the test report.

4.2.2.1 Mechanical preparation

The test piece shall be descaled mechanically by grinding on all surfaces and sharp edges, with grade 120 or finer, iron-free abrasive paper or cloth. Over-heating of the test piece shall be avoided.

4.2.2.2 Chemical preparation

The test piece without any mechanical treatment shall be descaled for no more than one hour, either in a solution of 50 volumes of hydrochloric acid ($\rho_{20} = 1,19$ g/ml), 5 volumes of nitric acid ($\rho_{20} = 1,40$ g/ml) and 50 volumes of water at 50 °C to 60 °C, or in a solution of 50 volumes of hydrochloric acid and 50 volumes of water at ambient temperature.

In the case of the chemical preparation, it shall be necessary to ensure in advance that intergranular corrosion does not occur due to the preparation. This should be achieved by micro examination of samples of each steel grade tested.

4.2.2.3 Degreasing

Immediately before being placed in the corrosive solution, the test piece shall be degreased in a suitable nonchlorinated agent, rinsed and dried.

5 Apparatus

See figure 1.

5.1 Erlenmeyer flask, of capacity at least 1 l, fitted with a cold finger immersion condenser or Erlenmeyer flask with a ground glass joint and equipped with an Allihn condenser with at least four bulbs. If an Allihn condenser is used it shall be checked by means of a paper indicator that no acid fumes are given off during the test.

For comparative measurements, the same type of apparatus shall be used for all the tests.

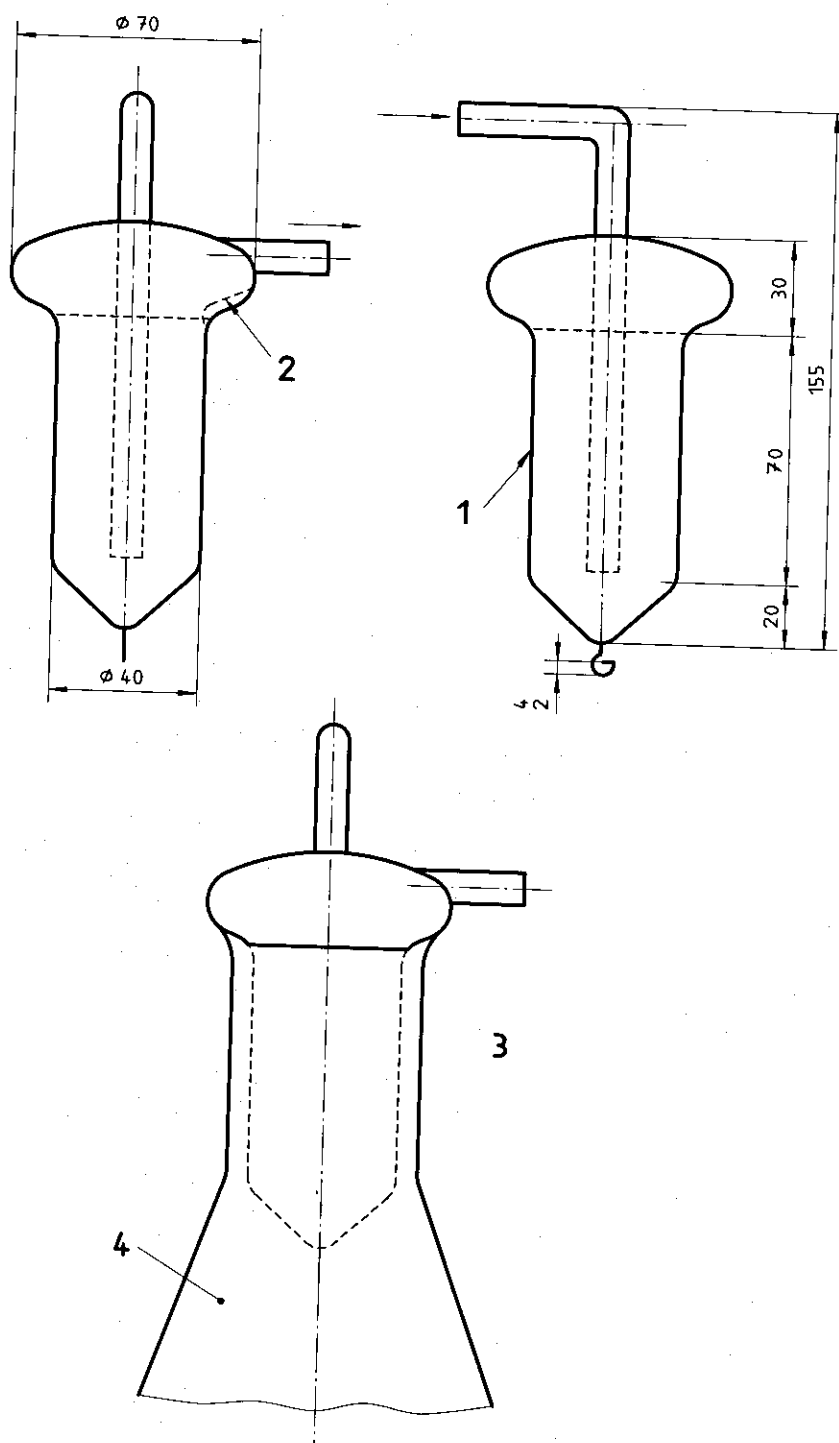
NOTE — It has been shown that corrosion rates obtained with a reflux condenser tend to be somewhat higher than with the cold finger type condenser due to greater vapour loss.

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

When several test pieces are treated in the same flask, the support shall be designed in such a way as to assure compliance with the requirement defined in 7.

5.3 Heating device, to keep the solution boiling.

Dimensions in millimetres

**Key**

- 1 Thickness of glass $\approx 1,5$ mm
- 2 2 or 3 notches (to avoid the joining by condensing)
- 3 Monitoring scheme of the condenser
- 4 Erlenmeyer flask

Figure 1 — Example of cold finger condenser

6 Corrosive solution

The corrosive solution shall be an aqueous solution of $65 \% \pm 0,2 \% (m/m)$ mass percent nitric acid ($\rho_{20} = 1,40 \text{ g/ml}$).

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

Solid residue $\leq 50 \text{ mg/kg}$

Pb $\leq 5 \text{ mg/kg}$

Fe $\leq 4 \text{ mg/kg}$

Mn negative test

As $\leq 1 \text{ mg/kg}$

Cl⁻ $\leq 3 \text{ mg/kg}$

SO₄²⁻ $\leq 50 \text{ mg/kg}$

PO₄³⁻ $\leq 10 \text{ mg/kg}$

F⁻ $\leq 3 \text{ mg/kg}$

7 Procedure

Use a volume of corrosive solution (see 6) of a least $20 \text{ ml}\cdot\text{cm}^{-2}$ 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. Excessive corrosion of one specimen may result in accelerated corrosion of the other specimens tested with it.

In case of dispute, only one test piece shall be placed in each flask. 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 boil. Boil for 5 periods of $48 \text{ h} \pm 1 \text{ h}$ each, using a fresh solution for each period, wash the test piece in water, dry and weigh. Care shall be taken to avoid excessive bubbling during boiling.

By special agreement between the interested parties, the number of test periods may be altered.

8 Calculation

The effect of the attack by the nitric solution is measured by determining the loss in mass of the specimen after each test period and for the total of the test periods.

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

$$r_{\text{corr}} = \frac{87\,600\,m}{S \cdot \rho \cdot t} \quad \dots (1)$$

$$r_{\text{corr}} = \frac{10\,000\,m}{S \cdot t} \quad \dots (2)$$

where

- t is the actual boiling period in hours;
- S is the original 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 for chromium nickel molybdenum steels and 7,9 g/cm³ for chromium nickel steels).

9 Test report

The test report shall contain at least the following information:

- a) reference to this part of ISO 3651, i.e. ISO 3651-1:1998;
- b) steel grade;
- c) heat treatment, if any;
- d) type of condenser used;
- e) mean corrosion rate;
- f) any incident which may have an effect on results.

Normally the average result for all the test periods is reported. Results from the individual periods may be reported by agreement between the interested parties.

ICS 77.060

Descriptors: iron and steel products, austenitic steels, stainless steels, tests, corrosion tests, intergranular corrosion tests, determination, corrosion resistance.

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