

INTERNATIONAL STANDARD

ISO
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Determination of resistance to intergranular corrosion of stainless steels —

Part 2:

**Ferritic, austenitic and ferritic-austenitic
(duplex) stainless steels — Corrosion test
in media containing sulfuric acid**

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

*Partie 2: Aciers inoxydables ferritiques, austénitiques et austéno-ferritiques
(duplex) — Essais de corrosion en milieux contenant de l'acide sulfurique*



Reference number
ISO 3651-2:1998(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 3651-2 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 3651-2:1976), 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*

Annexes A and B of this part of ISO 3651 are for information only.

Introduction

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

Ferritic, austenitic and ferritic-austenitic (duplex) stainless steels may be subject to such an 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 — In the field of application of this test, intergranular corrosion may be connected with the presence along the grain boundaries of a chromium-depleted region due to precipitation of chromium carbides, sigma phase or other intermetallic phases.

Determination of resistance to intergranular corrosion of stainless steels —

Part 2:

Ferritic, austenitic and ferritic-austenitic (duplex) stainless steels — Corrosion test in media containing sulfuric acid

1 Scope

This part of ISO 3651 specifies methods for the determination of the resistance to intergranular corrosion of ferritic, austenitic and ferritic-austenitic (duplex) stainless steels in media containing sulfuric acid. It also specifies the purposes which may be assigned to the test. The test methods included are:

- **method A:** the 16 % sulfuric acid/copper sulfate test (Monypenny Strauss test);
- **method B:** the 35 % sulfuric acid/copper sulfate test;
- **method C:** the 40 % sulfuric acid/ferric sulfate test.

The methods are applicable to stainless steels supplied in the form of cast, rolled or forged products and tubes and intended for use in a mildly oxidizing acid medium (for example, sulfuric acid, phosphoric acid).

Unless specified in the product standard, the method to be used, A, B or C, shall form the subject of an agreement between the interested parties.

Annex A gives examples of application of the three methods on stainless steels.

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. These test should, in no case, be considered as an absolute criterion of the quality of the metal.

2 Purpose of the test

This intergranular corrosion test may have either of the purposes given in 2.1 and 2.2.

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

This verification applies only to low carbon steels ($C \leq 0,03 \%$) and stabilized steels specified for resistance to intergranular corrosion. The metal is inspected after having undergone a heat treatment for sensitization which can be a heat treatment or welding 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. It is only of interest for the steels which are not defined in 2.1. The metal is inspected in the state in which it is delivered to the user, without heat treatment for sensitization.

3 Sensitization treatment

3.1 Sensitization heat treatment

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 low carbon content.

The sensitization heat treatment can be:

- T1: heating the specimen at $700\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$ for 30 minutes followed by water cooling;
- T2: heating the specimen at $650\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$ for 10 minutes followed by water cooling.

The above defined treatments only apply to austenitic and duplex steels.

The type of sensitization heat treatment shall be defined in the product standard or in the order. If not specified, heat treatment T1 shall be applied.

3.2 Sensitization by welding

Sensitization by the preparation of welded test pieces may be used as an alternative to 3.1, by agreement between the interested parties. Test pieces which are not solution annealed after welding shall be tested in the as-welded condition. No additional sensitization heat treatment shall be performed.

Sensitization by welding applies to all the stainless steels covered by this part of ISO 3651.

4 Corrosion test

4.1 Principle

A test piece, prepared as specified in 4.2 and 4.3, is immersed in a solution according to method A, B or C for a specified time. The test piece is then subjected to a bend test. The convex surface of the test piece is examined after bending in order to reveal any cracks caused by intergranular corrosion.

For small diameter tubes, up to 60 mm (tube diameter shall be compatible with the aperture of the flask containing the solution), a flattening test is used instead of the bend test.

4.2 Test pieces

4.2.1 Dimensions

The test piece shall have a total surface area of 15 cm^2 to 35 cm^2 . In the case of sheets with a thickness greater than 6 mm, the test piece shall have a maximum thickness of 6 mm and one of the rolled surfaces shall be retained.

The test pieces are selected from the product as defined in the product standard. In case of dispute, a flat test piece, when possible, shall be used having the following dimensions. These dimensions should be used in relation to the available bending apparatus:

- thickness between 2 mm and 6 mm;
- width at least 10 mm;
- length at least 50 mm.

4.2.2 Welded test pieces

Welded test pieces are prepared as follows.

For flat products, two pieces about 100 mm long and about 50 mm wide are welded together and the test piece is cut according to figure 1.

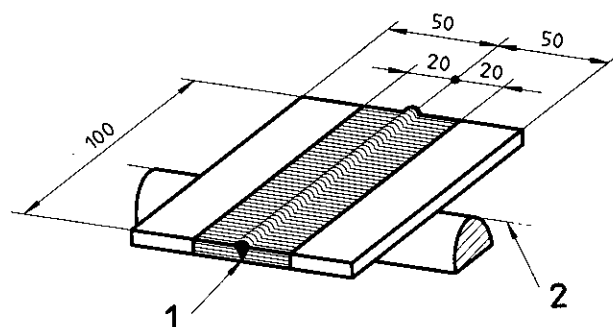
For tubes with a circumferential weld, the test piece is cut according to figure 2.

When four pieces are welded together to form a cross weld, the first-laid weld bead shall be in the longitudinal direction of the test piece as shown in figure 3.

If the thickness of the material is > 6 mm the test piece shall be machined down to 6 mm from one side. The retained surface shall form the convex side of the test piece after bending.

For welded tubes with outer diameter over 60 mm, the test pieces with length of at least 50 mm and width of at least 20 mm are prepared as shown in figure 4.

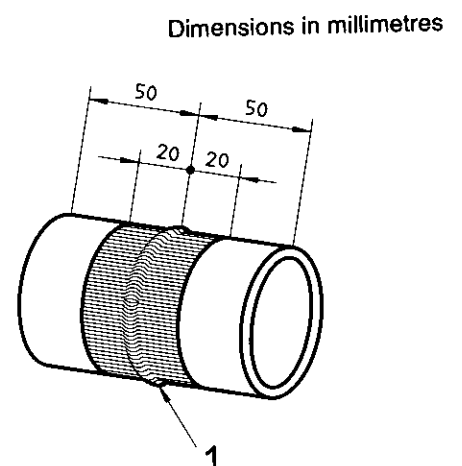
For welded tubes up to 60 mm outside diameter, the test piece shall be a full cross section of the tube and will be subject to a flattening test (see 6.4).



Key

- 1 Weld
- 2 Axis of the bending mandrel

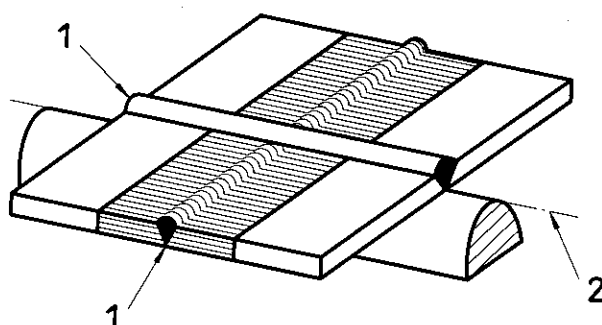
Figure 1 — Test piece for testing of plates and strips with welded butt joint



Key

- 1 Weld

Figure 2 — Test piece for testing of tube with welded butt joint

**Key**

- 1 Weld
- 2 Axis of the bending mandrel

Figure 3 — Test piece for testing of plates and strip with cross welded butt joint

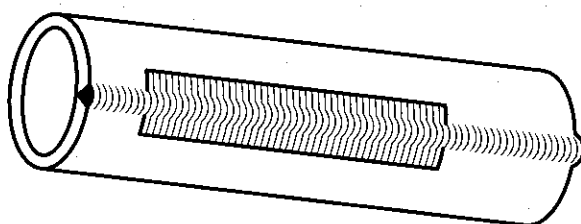


Figure 4 — Test piece for testing the longitudinal weld of welded tubes

4.3 Preparation of test pieces

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 either 4.3.1 or 4.3.2. Unless otherwise stated on the order, the method of preparation shall be left to the manufacturer and shall be recorded in the test report.

4.3.1 Mechanical preparation

The test piece shall be descaled mechanically by grinding in the longitudinal plane 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.

4.3.2 Chemical preparation

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

4.3.3 Degreasing

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

5 Apparatus

5.1 Erlenmeyer flask, capacity 1 l, or other suitable flask of optional capacity fitted, with an Allihn condenser with at least four balls.

5.2 Support for the test piece, generally of glass, for method C.

5.3 Heating device, to keep the solution boiling.

6 Test methods

6.1 Method A: The 16 % sulfuric acid/copper sulfate test (Strauss test)

6.1.1 Corrosive solution

The test solution shall be prepared as follows using analytical quality reagents.

Dissolve 100 g of copper (II) sulfate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in 700 ml of distilled water. Then add 184 g (100 ml) of sulfuric acid ($\rho_{20} = 1,84 \text{ g/ml}$) and make up to 1 000 ml with distilled water.

6.1.2 Procedure

More than one test piece may be tested in each flask provided that the volume of solution is at least $8 \text{ ml} \cdot \text{cm}^{-2}$ of the total surface area of the test pieces. The test pieces are embedded in electrical grade copper filings on the bottom of the flask. The amount of copper shall be at least 50 g/l of solution. The test pieces shall be in metallic contact with the copper but not with each other. The test pieces are firstly immersed in the cold test solution following which the solution is brought to the boil, from which point onwards the test duration commences. The solution shall be maintained at boiling $20 \text{ h} \pm 5 \text{ h}$. In case of dispute, the duration of the test shall be 20 h.

The copper filings may be reused if they are cleaned in warm tap water after each test. The corrosive solution shall be used only once.

6.2 Method B: The 35 % sulfuric acid/copper sulfate test

6.2.1 Corrosive solution

The test solution shall be prepared as follows using analytical quality reagents.

Add carefully 250 ml of sulfuric acid ($\rho_{20} = 1,84 \text{ g/ml}$) to 750 ml of distilled water. Dissolve 110 g copper (II) sulfate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in the warm solution.

6.2.2 Procedure

More than one test piece may be tested in each flask provided that the volume of solution is at least 10 ml·cm⁻² of the total surface area of the test pieces. The test pieces are embedded in electrical grade copper filings on the bottom of the flask. The amount of copper shall be at least 50 g/l of solution. The test pieces shall be in metallic contact with the copper but not with each other. The test pieces are firstly immersed in the cold test solution following which the solution is brought to the boil, from which point onwards the test duration commences. The solution shall be maintained at boiling during 20 h ± 5 h. In case of dispute, the duration of the test shall be 20 h.

The copper filings may be reused if they are cleaned in warm tap water after each test. The corrosive solution may be used more than once provided it is not discoloured and that its density has not changed.

6.3 Method C: The 40 % sulfuric acid/ferric sulfate test

6.3.1 Corrosive solution

The test solution shall be prepared as follows using analytical quality reagents.

Add carefully 280 ml of sulfuric acid ($\rho_{20} = 1,84$ g/ml) to 720 ml of distilled water. Dissolve 25 g ferric sulfate (III) $[\text{Fe}_2(\text{SO}_4)_3 \cdot x\text{H}_2\text{O}]$, containing about 75 % ferric sulfate) in the warm solution.

NOTE — Corrosion rates are expected to increase if all the ferric sulfate is used up.

6.3.2 Procedure

More than one test piece may be tested in each flask provided that the volume of solution is at least at 10 ml·cm⁻² of the total surface area of the test pieces. The test pieces are firstly immersed in the cold test solution following which the solution is brought to the boil, from which point onwards the test duration commences. The solution shall be maintained at boiling during 20 h ± 5 h. In case of dispute, the duration of the test shall be 20 h.

The test solution shall not be used more than once.

6.4 Bend test

Cylindrical and flat test pieces from wrought products shall be subject to a bend test after corrosion test. The test piece shall be bent to at least 90° over a mandrel with a radius not exceeding the thickness of the test piece. For cast products, the radius of the mandrel shall be no more than twice the thickness of the test piece.

Tubes with up to 60 mm outside diameter shall be subject to a flattening test. The distance between the platens after flattening, measured under load, shall be no greater than the following value, H , in millimetres:

$$H = \frac{1,09Dt}{0,09D + t}$$

where

t is the specified wall thickness, in millimetres;

D is the outside diameter of the tube, in millimetres.

Welded tubes shall have the longitudinal weld at the point of maximum strain except when the purpose of the test is to test a cross weld according to figure 2.

NOTE — A determination of the weight loss may be used as a complement to the bend test or the flattening test for tubes with up to 60 mm outside diameter according to 5.4. It is to be noted, however, that the weight loss will not measure the intergranular corrosion only but also any general corrosion occurring.

7 Evaluation

The bent test piece shall be examined under low magnification (about 10 ×), in order to detect cracking.

Cracking that originates at the edge of the specimen should be disregarded. The appearance of deformation lines, wrinkles, or "orange peel" on the surface, without accompanying cracks or fissures, should also be disregarded.

If it is questionable whether intergranular corrosion is the cause of the cracking, a 90 ° bend test or a flattening test shall be carried out on a second test piece prepared in a similar way but without having been submitted to the corrosion test. The comparison of the two pieces enables it to be established whether or not the cracks observed on the corroded test piece are due to intergranular corrosion.

NOTE — When an evaluation is questionable a metallographic examination of a longitudinal cross section of the test piece may be used to detect the presence of intergranular corrosion and to measure the depth of penetration.

8 Test report

The test report shall contain at least the following information:

- a) reference to this part of ISO 3651, i.e. ISO 3651-2:1998;
- b) steel grade;
- c) testing method applied (A, B or C);
- d) method of preparation (mechanical or chemical);
- e) nature of the test piece;
- f) nature of the sensitization heat treatment, when applied;
- g) results of the test;
- h) any incident which may have an effect on results.

Annex A (informative)

Examples of application

The method to be applied shall be specified in the product standard or defined by agreement. The following examples are given as information. Note that more than one method may be applicable for a specific steel grade.

Method A:

- austenitic steels with more than 16 % Cr and up to 3 % Mo;
- ferritic steels with 16 to 20 % Cr and 0 to 1 % Mo;
- duplex steels with more than 16 % Cr and up to 3 % Mo.

Method B:

- austenitic steels with more than 20 % Cr and 2 % to 4 % Mo;
- duplex steels with more than 20 % Cr and more than 2 % Mo.

Method C:

- austenitic steels with more than 17 % Cr and more than 3 % Mo;
- austenitic steels with more than 25 % Cr and more than 2 % Mo;
- ferritic steels with more than 25 % Cr and more than 2 % Mo;
- duplex steels with more than 20 % Cr and 3 % or greater Mo.

Annex B
(informative)

Comparison of commonly available national standards and this part of ISO 3651

National standard	Sensitizing treatment	Exposure duration	Angle of bending	Bending mandrel diameter	Examination magnification
prEN ISO 3651-2	700 °C for 30 min ¹⁾	20 h ²⁾	> 90°	$\leq t$	approx. 10 ×
ASTM A262:1993a	650-675 °C for 60 min	24 h	180°	t	5-20 ×
BS 5903:1980	unspecified	24 h	90°	t	$\leq 10 \times$
DIN 50914:1984	700 °C for 30 min	15 h	90°	4t	6-10 ×
NF A 05-159:1981	700 °C for 30 min (T1)	20 h ²⁾	90°	t	$\leq 10 \times$
SIS 117105:1977	unspecified	20 h	90°	t	10 ×
JIS G0575:1970	650 °C for 120 min	16 h	180°	approx. t	5-15 ×
<p>1) Proposed sensitization treatment (T1).</p> <p>2) It is acceptable, in certain circumstances, to extend the duration to 24 h.</p>					

ICS 77.060

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

Price based on 9 pages
