
**Corrosion of metals and alloys —
Accelerated corrosion test for
intergranular corrosion susceptibility
of austenitic stainless steels**

*Corrosion des métaux et alliages — Essai de corrosion accéléré pour
la détermination de la sensibilité à la corrosion intergranulaire
des aciers inoxydables austénitiques*



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Foreword

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Corrosion of metals and alloys — Accelerated corrosion test for intergranular corrosion susceptibility of austenitic stainless steels

1 Scope

1.1 This International Standard specifies accelerated methods for the determination of the intergranular corrosion susceptibility of austenitic stainless steels. The methods specified by the standard provide results identical to those obtained when using ISO 3651-1 and ISO 3651-2, but the test period for the accelerated corrosion test is shorter.

1.2 This International Standard is applicable to the testing of various kinds of metal production, including two-layer rolled metal, welded joints, deposited metal and weld seam metal.

1.3 Two test methods are specified:

- method A: corrosion test in a solution of copper sulfate and concentrated sulfuric acid in the presence of metallic copper;
- method B: corrosion test in a solution of copper sulfate, sulfuric acid and copper fluoride in the presence of metallic copper.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3651 (all parts), *Determination of resistance to intergranular corrosion of stainless steels*

ISO 8044, *Corrosion of metals and alloys — Basic terms and definitions*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 8044 apply.

4 Test specimens

4.1 Test specimens may be taken from

- any position within steel plates with a width equal to or less than 10 mm, or from strip or wire,
- the axis zone (in the longitudinal direction) of rolled metal (rolled section metal and shaped rolled metal with a round, square or hexagonal cross-section),

- any convenient position in other kinds of rolled metal,
- the axis zone (in the longitudinal or cross-sectional direction) of tube samples,
- any convenient position in tubes,
- the body or spare material from forgings, or
- the bosses of casts or specially cast test pieces.

4.2 Test specimens should be produced from samples of various types, as indicated in Table 1.

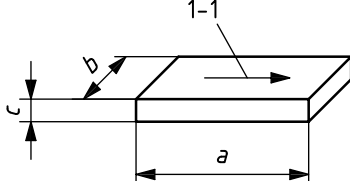
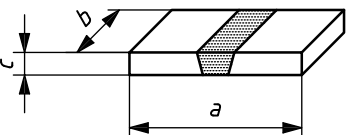
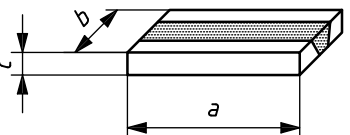
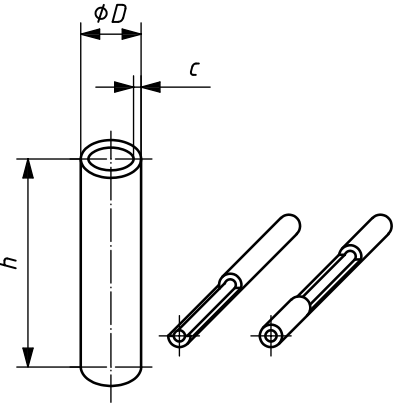
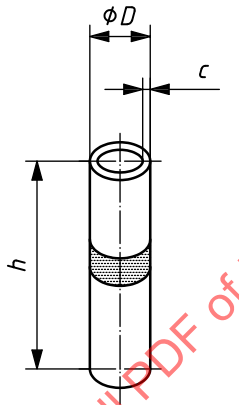
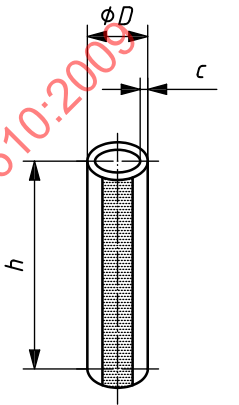
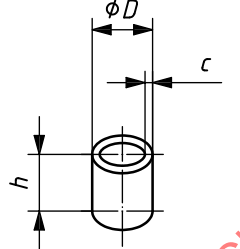
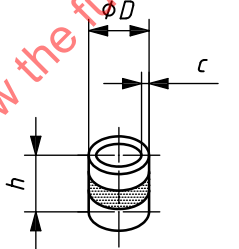
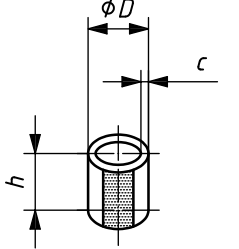
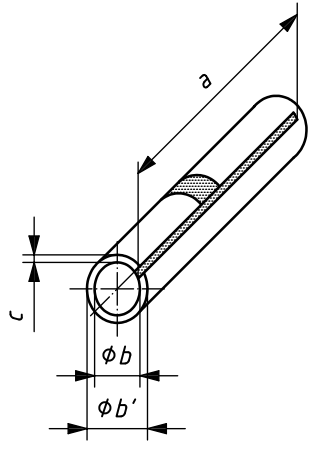
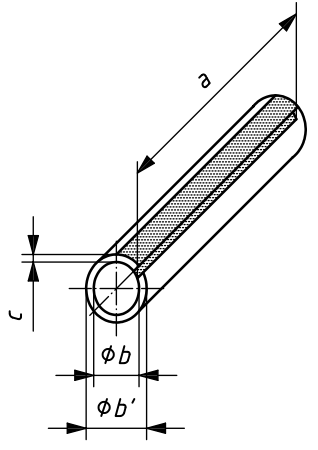
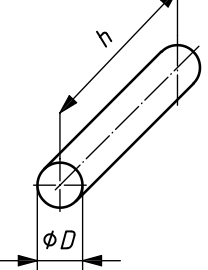
Table 1 — Types of specimen

Types of sample	Test specimens
Steel plates, strip, wire, rolled section or shaped rolled metal, tube samples, forged pieces, cast pieces, metal from weld seams	Flat (see Table 2)
Cylindrical with a diameter of not less than 10 mm	Cylindrical (see Table 2)
Tubes	Flat, segments, rings ($D > h$), tubular ($D < h$) (see Table 2), where D is the external diameter and h is the height

It is permissible to

- subject rings and tubular samples with a specified wall thickness of not less than 1,5 mm to reaming, straightening or flattening and subsequent cutting of the sides with the purpose of producing flat specimens,
- remove the wall of a tubular specimen for 1/2 of its circumference from one of its ends or in the middle of its length for 1/2 of the overall length (see Table 2), and
- manufacture specimens from rolled clad plate or bimetallic tubes in the same way as from flat sheet products and ordinary tubes, respectively (specimens may be produced from the basis layer following the removal of the other layer and interfacial region by machining).

Table 2 — Types of specimen

Specimen type	Specimen without welding	Specimen with welding	
		Type 1	Type 2
Flat			
Tubular			
Ring			
Segment			
Cylindrical			

4.3 For the purpose of revealing cracks, the specimen size should allow the following:

- a) flat specimens, segment specimens, cylindrical specimens with a diameter of no more than 5 mm and tubular specimens with a diameter of no more than 5 mm to be bent at an angle of $(90 \pm 3)^\circ$;
- b) ring and tubular specimens with a diameter of more than 5 mm to be flattened and formed into a cone;
- c) examination of the surface of the bend in a section with sufficient thickness to provide reliable recognition of intercrystalline cracks.

4.4 Specimens of the required thickness are produced from the blanks by machining.

- a) For metal plates, the specimens should be taken from only one side; if one of the plate sides has been treated, the specimen should be taken from the treated side.
- b) For rolled metal (rolled section and shaped), forgings, castings and tube samples the specimens should be taken from any convenient surface.
- c) For tubes after hot or cold deformation, the specimens should be taken from the outside.
- d) For hot-rolled tubes, the specimens should be taken from either the inner or outer surface.
- e) For tubes that have been exposed to operating conditions, the specimen should be taken from the side that was in contact with the service environment.

4.5 The following types and kinds of specimen should be produced out of welded joints (see Table 2).

- Flat specimens of types 1 or 2 should be produced out of joint welds of plate, section or shaped rolled metal, cast pieces and forged pieces.
- Ring, tubular and segment specimens of type 2 should be produced out of electrically welded tubes.
- Segment, ring or tubular specimens of type 1 should be produced out of ring welded joints of tubes.
- Flat specimens of types 1 or 2, and segment, ring or tubular specimens of type 1, should be produced out of welded joints of two-layer tubes.

4.6 For welded specimens (Table 2, types 1 and 2), the reinforcement of the welded seam should be removed by machining to a depth not exceeding 1 mm.

The desired thickness of the specimen should be achieved by means of machining, and the metal should be removed from the side that was not in contact with corrosive medium. If it is not known which side was in contact with the corrosive medium, the machining should be carried out on the side where the metal of the welded seam was exposed to the least heating during welding.

The wall thickness of welded tubular specimens should be decreased in accordance with 4.4.

4.7 Coated electrodes, filler wire and tape should be checked by means of tests on deposited metal or weld seam metal produced from these materials.

Deposited metal should be tested through the use of flat specimens (see Table 2). The specimens should be cut out of multi-layer deposits made from checked welding materials (the bottom layers of the deposits, which are not used for test specimen production, could be made from different materials of similar chemical composition). Where the chemical composition of the plate on which the metal is being deposited is similar to the chemical composition of the deposited metal, the number of the lower layers, which should not be used for specimen production, may be decreased to three.

Metal from the weld seam should be tested using flat specimens, which should be cut from the upper layers of the multi-layer seam of the welded joint being checked. The seam thickness in the zone from which the

specimen is cut out should not be less than 15 mm. The plates used for the welded joint should be of steel with a chemical composition similar to that of the weld metal.

When thin plates are being used to provide the desired weld seam thickness, special weld backing may be used. This is produced from steel of chemical composition similar to that of the plate. (Where preliminary additions are to be made to the edges of thin plate specimens, similar additions should be made to the whole surface of the weld backing.)

The metal of the weld seam should be tested through the use of flat welded specimens (see Table 2, types 1 and 2) which are produced from control-welded joints made of steel that is resistant to intergranular corrosion, and preferably using filler materials intended specially for welding the steel in question.

4.8 The recommended sizes of the specimens are indicated in Annex A. However, it is also permissible to use different specimen sizes.

4.9 For the purpose of testing plate, tubes, metal sections, wire and tape, a set of at least two test specimens should be produced.

Not less than four test specimens (two of which should be for reference purposes) should be produced from each forging, casting and weldment for all types of steel and alloy of interest.

Control specimens should be bent to an angle of $(90 \pm 3)^\circ$ and should not be exposed to the boiling test solution. If it is necessary to check the specimens from both sides, they should be bent into a Z-shape.

4.10 Sensitization treatment

4.10.1 Non-stabilized steels

Non-stabilized steels should be tested using specimens in the as-delivered condition provided they do not contain titanium or niobium, provided they have a specified maximum carbon content of more than 0,030 % and provided they are to be used in the as-delivered condition, unless there are indications to the contrary in the accompanying technical documentation.

4.10.2 Stabilized steels

4.10.2.1 Stabilized steels and alloys containing titanium or niobium with specified maximum carbon contents not in excess of 0,030 %, if to be used as-delivered, should be tested using specimens made from metal taken from the production line and then subjected to heat treatment at up to $(700 \pm 10)^\circ\text{C}$ for 30 min with subsequent cooling in water (see ISO 3651-1 and ISO 3651-2).

A different sensitization heat treatment may be carried out with the agreement of the customer.

Either the samples from which the specimens are made or the specimens themselves may be given the sensitization heat treatment.

If discrepancies arise in the interpretation of the test results, the samples should be subjected to a sensitization heat treatment procedure. To do so, degreased samples should be placed in a furnace which has been pre-heated to the sensitization temperature.

4.10.2.2 Steels which are to be used in a cold-worked state or in a semi-cold-worked state should be tested using specimens that have not been given a sensitization heat treatment.

4.10.2.3 At companies which manufacture welded joints, test specimens from weldments can be used without sensitization heat treatment if the welding conditions have been properly controlled.

4.10.2.4 Where steels and alloys are to be tested after repeated heat treatment which differs from the sensitization heat treatment, testing should be carried out in accordance with this subclause (4.10) as for the testing of a new batch of metal.

4.10.2.5 In cases when, during the manufacture of an item, control-welded joints are subjected to heat treatments that can influence their properties, the test specimens should be treated under similar conditions.

4.10.2.6 Scale that forms on the test specimen surface during hardening or sensitization heat treatment should be removed by chemical or electrochemical etching or by a mechanical process before grinding and polishing. The depth of any surface treatment should not exceed 1 mm.

Chemical etching should be carried out either in the following solution:

- (620 ± 3) ml of HNO₃ (ρ_{20} = 1,35 g/ml);
- (76,0 ± 0,1) g of NH₄F;
- water (300 ± 3) ml;
- temperature (20 ± 5) °C;

or electrolytically as follows:

- (34 ± 1) ml of orthophosphoric acid (ρ_{20} = 1,68 g/ml);
- (11 ± 1) ml of HNO₃ (ρ_{20} = 1,35 g/ml);
- water (955 ± 3) ml;
- temperature 40 °C to 50 °C;
- current density $0,5 \times 10^4$ A·m⁻² to $0,6 \times 10^4$ A·m⁻².

The test specimen should be etched to completely remove any scale. After etching, the specimens should be carefully rinsed with water.

Other etching solutions and etching regimes may be used for scale removal if they provide complete scale removal and, for steels that are resistant to intergranular corrosion, also prevent both preferential etching along the grain boundaries and pitting corrosion.

Where discrepancies arise in the test results, etching should be carried out only with the solutions prescribed above.

4.11 The roughness of the controlled surfaces of test specimens prior to testing should not exceed 0,8 µm. The required finish should be achieved by polishing or grinding. Overheating of the surface should be avoided.

Test specimens which are produced from tubes that have been subjected to cold or hot deformation or from cold-rolled or cold-drawn metal products or from metal products with special surface treatments should not be subjected to polishing or grinding unless this is called for in the technical documentation.

4.12 Specimens should be marked before testing. The identification marks should be applied either by stamping or by electrical discharge machining of brittle material at one or both ends of the specimen at a distance of 5 mm to 10 mm from the edge.

4.13 Before testing, the specimens should be degreased using suitable organic solvents of high quality (reagent grade). If the specimens are placed in the test vessel directly after the completion of etching or rinsing procedure, the degreasing procedure may be omitted.

5 Test methods

5.1 Method A

5.1.1 Principle of the method

The specimens of steel together with metallic copper should be placed into an aqueous solution of H_2SO_4 and $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ of higher concentrations than for the method specified in ISO 3651-2.

The proposed method is an accelerated one and should be used for verifying the grades of steel considered in ISO 3651-2.

5.1.2 Test solution

The test solution is prepared as follows, using analytical-grade reagents: $(50 \pm 0,1)$ g of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ is added to $(1\,000 \pm 3)$ ml of water, then (250 ± 3) ml of H_2SO_4 ($\rho_{20} = 1,84$ g/ml) is added in small portions.

5.1.3 Sequence of test steps

5.1.3.1 The test is carried out in a glass flask fitted with a condenser or in a vessel made of corrosion-resistant stabilized chromium-nickel steel with a cover and a condenser. A layer of copper filings should be placed on the bottom of the reaction vessel, with the specimens on top of it.

It is permissible to do the following:

- a) Use suspended copper plates with holes of diameter 5 mm to 7 mm having sharp edges instead of copper filings where reliable contact with both the top and bottom of the specimens can be assured in order to ensure good rinsing of the specimens by the test solution and the reliable removal of corrosion products. Should discrepancies occur in the assessment of metal quality, the test should be carried out in the presence of copper filings.
- b) Expose several rows of specimens where there is reliable contact of the specimens (from both sides) with the copper filings or reliable two-sided contact between the specimens and the copper plates. The level of the test solution should be 20 cm higher than the upper level of the test specimens or the copper filings or the copper plates.

After the solution has been poured into the vessel, it is heated to its boiling point and boiled as specified in 5.1.3.3.

5.1.3.2 Heating of the condenser should be avoided.

5.1.3.3 The time of boiling should be $(8,00 \pm 0,25)$ h. In the case of a forced interruption of testing, the test specimens should not be removed from the test solution. The duration of the test is determined as the sum of all boiling periods.

5.1.3.4 If discrepancies occur in the results of interrupted tests, a repeat test should be carried out with continuous exposure to boiling conditions in the glass flask. In this case, the solution volume should be not less than 10 ml per 1 cm² of specimen surface.

5.1.3.5 After testing in the boiling solution is over, the specimens should be rinsed with tap water and dried. In cases where a copper layer which cannot be washed off with water is deposited on the specimen surface, the copper layer should be removed by rinsing with a 20 % to 30 % nitric acid solution.

5.1.3.6 The repeated use of the test solution is permitted provided that the colour of the solution does not change. The only exception to this is where there is disagreement between the parties interested in the results of the tests.

5.1.3.7 Copper filings or copper plates may be used repeatedly. Should surface darkening occur during storage, they should be rinsed in 20 % to 30 % nitric acid solution and then in tap water.

5.2 Method B

5.2.1 Principle of the method

The specimens of steel should be placed in an aqueous solution of CuSO_4 , H_2SO_4 and NaF, in the presence of metallic copper.

5.2.2 Test solution

Using analytical-grade reagents, $(50 \pm 0,1)$ g of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and $(128 \pm 0,1)$ g of NaF are dissolved in $(1\,000 \pm 3)$ ml of water, then (250 ± 1) ml of H_2SO_4 ($\rho_{20} = 1,84$ g/ml) is added in small portions to prevent overheating of the solution. The $(128 \pm 0,1)$ g of NaF may be replaced by $(177 \pm 0,1)$ g of KF. The solution should be prepared in a polyethylene vessel.

5.2.3 Sequence of test steps

5.2.3.1 The test is carried out in a polyethylene vessel, covered by a lid, at a temperature between 20 °C and 30 °C, but otherwise in accordance with 5.1.3.1.

5.2.3.2 The duration of the test is $(2 \pm 0,1)$ h for steel which does not contain molybdenum and $(3 \pm 0,1)$ h for steel which contains molybdenum.

5.2.3.3 After the test, the specimens are treated in accordance with 5.1.3.5.

5.2.3.4 Both the test solution and the metallic copper may be used repeatedly in accordance with 5.1.3.6 and 5.1.3.7.

6 Evaluation of intergranular corrosion

6.1 To reveal any intergranular corrosion after testing in the test solution, the specimens should be bent through an angle of $(90 \pm 3)^\circ$.

Table 3 gives suitable radii for arbors around which specimens taken from various kinds of metal product may be bent.

6.2 Where the exposed surface of a specimen that has been exposed in service is not identified, both sides of the specimen should be checked after testing. For this purpose, the specimen is bent into a Z-shape.

When specimens made from rolled clad plate or bimetallic tubes are being tested, they should be bent in such a way that the surface of the specimen from which material has been removed is on the inside.

Table 3 — Bending radii of specimens

Kinds of metal product	Radii of arbors to be used for bending specimens
Plate, rolled section metal, tubes, forgings	1 mm for specimens with thicknesses of not more than 1 mm. Not more than the specimen thickness for specimens with thicknesses in excess of 1 mm.
Castings, welded joints, weld seam metal, built-up weld metal	2 mm for specimens with thicknesses of not more than 1 mm. Not more than twice the specimen thickness for specimens with thicknesses greater than 1 mm but not more than 3 mm. 10 mm for specimens with thicknesses in excess of 3 mm.

6.3 Flat and segment specimens should be bent into a Z-shape if they are made from seamless tubes or cut out of straightened or flattened rings or tubes.

If specimens cannot easily be bent into a Z-shape, twice the number of specimens should be used for the test. Half of them should be bent from the convex side and the other half from the concave side.

The external surface of specimens made from seamless tubes with an external diameter of not more than 5 mm should be assessed after bending. The internal surface should be assessed either metallographically or, in cases where part of the tube wall has been removed (see 4.2), by bending.

Ring and tubular specimens made from seamless tubes with diameters in excess of 8 mm should be assessed by flattening them until the separation of the compressing surfaces is H mm, where the value of H is calculated from Equation (1):

$$H = 1,08Dc/(0,08D + c) \quad (1)$$

where

D is the external tube diameter, in millimetres;

c is the wall thickness, in millimetres.

The internal surfaces of ring and tubular specimens should be assessed metallographically. In the case of austenitic steels, the assessment of internal ring surfaces may be carried out by "beading" (i.e. locally reducing the diameter) to a value which is calculated from Equation (2):

$$d = 1,56c \quad (2)$$

where d is the internal ring diameter, in millimetres.

6.4 After testing, welded specimens should be bent in accordance with 6.1.

a) Type 1 specimens (see Table 2) should be bent

- 1) along the weld when the weld metal is being assessed;
- 2) along the heat-affected zone when the parent steel is being assessed.

When the weld joint as a whole is being assessed, some of the specimens should be bent along the weld and some along the heat-affected zone.

b) Type 2 specimens (see Table 2) should be bent

- 1) transversely when the weld metal, the heat-affected zone or the weld joint as a whole is being assessed;
- 2) into a Z-shape when both parts of the specimen are being assessed.

6.5 In order to reveal weld boundaries, it is permissible to etch specimens lightly for 0,5 min to 1 min using one of the solutions specified in 4.10.2.6.

6.6 The bend should be made in such a way that the weld surface that is normally in contact with the operating environment is located on the outside of the specimen. If it is not known which side of the specimen is in contact with the operating environment, then the side of the weld which had the most heating should be on the outside of the specimen.

6.7 In cases when the specimen surface has been mechanically processed, the specimen should be bent away from the side which was not processed.

Ring and tubular specimens with longitudinal or circumferential weld seams should be flattened in accordance with 6.3. In such cases, the ring seam should be placed along the direction of the flattening load and the longitudinal seam should be placed at right angles to the direction of the flattening load and in the diametrical plane.

6.8 The bent specimens should be examined using a magnifying glass with a magnification of 8 to 12 times.

6.9 When cracks are not found in specimens which have been bent after testing (apart from longitudinal cracks and cracks directly on the edges), the material is considered to be resistant to intergranular corrosion. However, if cracks are found, testing may be repeated, this time in accordance with ISO 3651-2.

6.10 In cases when, after exposure in the test solution and during or after bending,

- a) the control specimens break or
- b) cracks are found in specimens which were bent after testing (apart from longitudinal cracks and cracks directly on the edges) or
- c) the specimen thickness is less than 0,1 mm and intergranular corrosion is not found or
- d) it is impossible to bend the specimen because of its size,

then the intergranular corrosion susceptibility should be evaluated by metallography.

6.11 Before metallographic evaluation of the intergranular corrosion susceptibility of specimens that have undergone corrosion testing, the specimen for metallographic examination should be cut out of the corrosion-tested specimen along a cutting plane perpendicular to the specimen surface under assessment.

In the case of metallographic specimen preparation from welded specimens, the cutting line should be perpendicular to the weld line and the cutting plane should include both the weld metal and the metal of the heat-affected zone.

The recommended metallographic specimen length from the surface to be assessed should be 15 mm to 20 mm.

The cut surface should be coincident with the metallographic specimen surface.

The metallographic specimen should be produced in such a way that no corner breaks or fins are generated.

6.12 To reveal intergranular corrosion and evaluate the depth of any corrosion-related cracking, previously etched metallographic specimens should be examined at a magnification of 200 times. The etching should produce only slight attack of the grain boundaries.

A list of recommended etching reagents and procedures is given in Clause A.2.

The surfaces under investigation of the metallographic specimens should be examined.

The maximum depth of attack should be evaluated for six different fields of view. The sites with the greatest depths of intergranular corrosion should be included in these fields of view.

6.13 The material may be reported as being resistant to intergranular corrosion if the metallographic assessment reveals the maximum depth of corrosion attack to be not more than 30 µm, unless the specifications for the metal products contain other requirements. For specimens produced from metal products with thicknesses of less than 1,5 mm, the maximum depth of corrosion should be not more than 10 µm.

7 Test report

The following information should be included in the test report:

- a) the steel grade;
- b) the type of metal product;
- c) the method of manufacture of the product;
- d) the heat treatment used;
- e) the test specimen type and size;
- f) the test method used;
- g) the result of the test, i.e. whether the specimens are resistant to intergranular corrosion or not resistant to intergranular corrosion.

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Annex A

(informative)

Recommended standard specimen sizes and recommended chemical and electrolytic cleaning procedures for removal of corrosion products

A.1 Recommended standard specimen sizes

Table A.1 — Standard specimen sizes

Material		Standard specimen sizes mm				
		A	B	C	D	H
Steel plates of thickness less than 10 mm		Not less than 50	$20,0 \pm 0,5$	As for blank	—	—
Wire, rolled section or shaped rolled metal of thickness or diameter in excess of 10 mm		Not less than 50	As for blank	As for blank	As for blank	—
Steel plates of thickness in excess of 10 mm		Not less than 50	$20,0 \pm 0,5$	3 to 5	—	—
Forged pieces, cast pieces, rolled section or shaped rolled metal of thickness or diameter in excess of 10 mm		Not less than 50	10 to 20	3 to 5	—	—
Steel plates of thickness in excess of 10 mm		Not less than 50	$20,0 \pm 0,5$	Not more than 5	—	—
Seamless tubes of diameter in excess of 5 mm:	flat or segment specimens	Not less than 50	Not more than 20	Not more than 5	—	—
	ring or tubular specimens	—	—	Not more than 5	Not more than 40	Not less than 0,5
Seamless tubes of diameter less than 5 mm		Not less than 80 Cut wall at 1/2 of height and over a length of 35 to 40	—	—	—	—
Seamless bimetallic tubes:	flat or segment specimens	Not less than 50	Not more than 20	Not more than 5	—	—
	ring or tubular specimens	—	—	Not more than 5	Not more than 40	Not less than 0,5