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Corrosion of metals and alloys — Removal of corrosion products from corrosion test specimens

Corrosion des métaux et alliages — Élimination des produits de corrosion sur les éprouvettes d'essai de corrosion

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Foreword

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The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 8407 was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

This second edition cancels and replaces the first edition (ISO 8407:1991), which has been technically revised.

Corrosion of metals and alloys — Removal of corrosion products from corrosion test specimens

WARNING — Safety rules for personnel: handling of the solutions used for removal of corrosion products must be left to skilled personnel or conducted under their control. The equipment must be used and maintained by skilled personnel, not only so that the procedures can be performed correctly, but also because of the hazards to health and safety that are involved.

1 Scope

This International Standard specifies procedures for the removal of corrosion products formed on metal and alloy corrosion test specimens during their exposure in corrosive environments. For the purpose of this International Standard, the term "metals" refers to pure metals and alloys.

The specified procedures are designed to remove all corrosion products without significant removal of base metal. This allows an accurate determination of the mass loss of the metal, which occurred during exposure to the corrosive environment.

These procedures may, in some cases, also be applied to metal coatings. However, possible effects from the substrate must be considered.

2 Procedures

2.1 General

2.1.1 A light mechanical cleaning treatment by brushing with a soft bristle brush under running water should first be applied to remove lightly adherent or bulky corrosion products.

2.1.2 If the treatment described in 2.1.1 does not remove all corrosion products, it will be necessary to use other procedures. These are of three types:

a) chemical;

b) electrolytic;

c) more vigorous mechanical treatments.

NOTE These treatments will also remove some base metal.

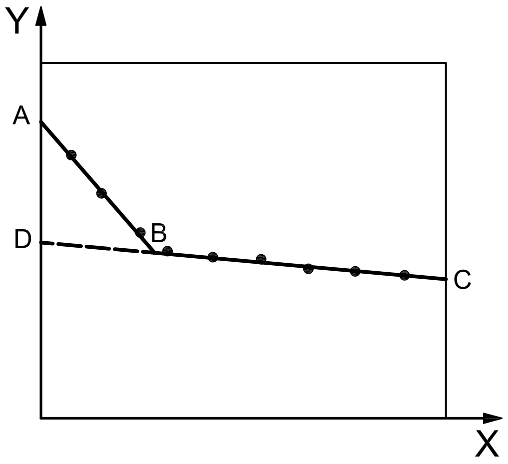
Whichever method is used, it might be necessary to repeat the cleaning treatment to ensure complete removal of the corrosion products. Removal shall be confirmed by visual examination. The use of a low-power microscope (i.e. ×7 to ×30) is particularly helpful with a pitted surface since corrosion products may accumulate in pits.

2.1.3 An ideal procedure should remove corrosion products and not result in removal of any base metal. Two procedures can be used to confirm this point. One procedure uses a control specimen (2.1.3.1) and the other requires a certain number of cleaning cycles on the corroded specimen (2.1.3.2).

2.1.3.1 Uncorroded control specimens, which should be similar chemically, metallurgically, and geometrically to the test specimens, should be cleaned by the same procedure as used for the test specimen. By weighing the control specimen before and after cleaning (weighing to the fifth significant figure is suggested, e.g. a 70 g specimen should be weighed to three decimal places), the metal loss resulting from cleaning may be determined. The mass loss of the control specimen will reflect the mass loss of test specimens resulting from the cleaning procedure.

2.1.3.2 The cleaning of each corroded test specimen should be repeated several times after the removal of the corrosion products is completed. The mass shall be plotted as a function of the number of equal cleaning cycles (see Figure 1). Point A represents the mass of corroded specimens before the start of the cleaning. In many cases two straight lines, AB and BC, will be obtained. Line AB characterizes the removal of corrosion products and may not always be visible. Line BC characterizes the removal of substrate after the corrosion products are gone. Point D, which characterizes the mass of the pure metal at zero number of cleaning cycles, is obtained by extrapolation of line BC to the ordinate axis. In some cases, the relation may not be linear and the most appropriate extrapolation shall then be made.

If no intervals of equal cleaning cycles are used during one cleaning procedure, the x-axis should be expressed in time units.



Key

X numbers of cleaning cycles or time units

Y mass

Figure 1 — Mass of corroded specimens after repetitive cleaning cycles

2.1.3.3 The true mass of the specimen, after removal of the corrosion products, will be a value between the masses represented by the points B and D, depending on the degree of protection furnished by the corrosion products during the cleaning procedure.

2.1.4 The preferred cleaning method will be that which

a) provides efficient removal of corrosion products,

b) provides low or zero mass loss when applied to new uncorroded specimens (see 2.1.3.1), and

c) provides a curve of mass as a function of the number of cleaning cycles or time of pickling, which is close to horizontal when the latter is plotted as the abscissa (see 2.1.3.2).

2.1.5 When chemical or electrolytic procedures are used, solutions freshly prepared with distilled or deionized water and reagent grade chemicals shall be used.

2.1.6 After cleaning, the specimen should be thoroughly rinsed with tap water, a light brushing during this procedure will help to remove any remaining surface products resulting from the cleaning process. Finally, the specimens shall be rinsed with distilled or deionized water. The specimen shall then be rinsed thoroughly in ethanol and dried using a hot air blower or an oven. After that, the specimens shall be allowed to cool in a dessicator to the balance room temperature before weighing.

2.2 Chemical procedures

2.2.1 Chemical procedures involve immersion of the corrosion test specimen in a chemical solution which is specifically designed to remove the corrosion products with minimal dissolution of any base metal. Several procedures are listed in Annex A (see Table A.1). To facilitate the cleaning, it is strongly recommended to use an ultrasonic bath treatment.

2.2.2 Chemical cleaning is often preceded by light brushing of the test specimen to remove lightly adherent, bulky corrosion products.

2.2.3 Before the chemical treatment, clean the specimens as described in 2.1.1. In connection with the intermittent removal of specimens for weighing, brush the specimens, if necessary, to facilitate the removal of tightly adherent corrosion products.

2.3 Electrolytic procedures

Electrolytic cleaning can also be used to remove corrosion products. Several methods of electrolytic cleaning of corrosion test specimens are given in Annex A (see Table A.2). To facilitate the cleaning, it is strongly recommended to use an ultrasonic bath treatment.

Electrolytic cleaning should be preceded by light brushing of the test specimen to remove lightly adherent, bulky corrosion products. Brushing should also follow electrolytic cleaning to remove any loose slime or deposits. This will help to minimize any redeposition of metal from reducible corrosion products, which would reduce the mass loss.

2.4 Mechanical procedures

Mechanical procedures can include scraping, scrubbing, brushing, ultrasonic methods, mechanical shock and impact blasting (grit blasting, water-jet blasting, etc.). These methods are often used to remove heavily encrusted corrosion products. Scrubbing with a bristle brush and a mild abrasive/distilled water slurry can also be used to remove corrosion products.

Vigorous mechanical cleaning will result in the removal of some base metal, so care should be exercised. These methods should be used only when others fail to provide adequate removal of corrosion products. As with the other methods, correction for metal loss due to the cleaning method is recommended. The mechanical forces used in cleaning shall be maintained as constant as possible.

3 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) the procedure used to remove corrosion products;

c) for chemical procedures, the composition and concentration of the chemicals employed or the designation according to Table A.1, the solution temperature and the duration of each cleaning cycle or number of equal cleaning cycles;

d) for electrolytic procedures, the composition and concentration of chemicals employed or the designation according to Table A.2, the solution temperature, the anode material and current density, and the duration of cleaning;

e) for mechanical procedures, the specific mechanical method employed (bristle-brush scrubbing, wooden scraper, etc.), abrasive compounds used, and the duration of cleaning;

f) where multiple procedures are used, the appropriate details for each method and the sequence of methods;

g) the results of control cleaning (see 2.1.3.1) or from repetitive cleaning cycles (see 2.1.3.2) designed to assess mass loss from the removal of base metal during the cleaning process;

h) the mass loss due to corrosion (see 2.1.3.3).

Annex A
(informative)

Chemical and electrolytic cleaning procedures for removal of corrosion products

A.1 General

In the development of this International Standard, a number of sources were consulted to identify chemical and electrolytic cleaning procedures. This annex summarizes the results of this survey.

Prior to adopting these cleaning procedures, the user should conduct a test on control samples to ensure the efficiency of the chosen method.

A.2 Procedures

Tables A.1 and A.2 summarize various chemical and electrolytic cleaning procedures for removal of corrosion products. The specific choice of procedure for a given material will depend on many factors, including previous experience. If more than one chemical or electrolytic cleaning procedure is given for a metal, the procedures are usually listed in preferred order. This International Standard should be consulted for guidance in the proper application of the procedures in Tables A.1 and A.2.

For all the cleaning procedures listed, it is recommended that the surfaces should be maintained vertical during cleaning. This will minimize retention of any gases released during the cleaning procedure.

The times specified represent recommendations appropriate for mass-loss studies in the context of Figure 1. However, when the cleaning procedure is adopted for other purposes (e.g. cleaning of fracture surfaces for inspection, using a microscope), the exposure time may be longer.

WARNING — When working with dangerous substances (such as cyanide, chromium trioxide, zinc dust), all necessary safety precautions shall be taken.

Table A.1 — Chemical cleaning procedures for removal of corrosion products

Designation	Material	Chemical products	Total time	Temperature	Remarks
C.1.1	Aluminium and aluminium alloys	Nitric acid (HNO_3, $\rho = 1,42$ g/ml)	1 min to 5 min	20 °C to 25 °C	To avoid reactions that may result in excessive removal of base metal, remove extraneous deposits and bulky corrosion products.
C.1.2[a]		50 ml of phosphoric acid (H_3PO_4, $\rho = 1,69$ g/ml) 20 g of chromium trioxide (CrO_3) Distilled water to make 1 000 ml	5 min to 10 min	80 °C	If corrosion-product films remain, then follow with the nitric acid procedure as for designation C.1.1.
C.2.1	Copper and copper alloys	50 g of amidosulfonic acid (sulfamic acid) Distilled water to make 1 000 ml	5 min to 10 min	20 °C to 25 °C	
C.2.2		500 ml of hydrochloric acid (HCl, $\rho = 1,19$ g/ml) Distilled water to make 1 000 ml	5 min to 10 min	20 °C to 25 °C	De-aeration of solution with purified nitrogen will minimize base metal removal.

Table A.1 (*continued*)

Designation	Material	Chemical products	Total time	Temperature	Remarks
C.2.3		54 ml of sulfuric acid (H_2SO_4, $\rho = 1{,}84$ g/ml) Distilled water to make 1 000 ml	1 min to 10 min	20 °C to 25 °C	De-aerate solution with nitrogen. Brushing of test specimens to remove corrosion products followed by re-immersion for 3 s to 4 s is recommended.
C.3.1	Iron and steel	500 ml of hydrochloric acid (HCl, $\rho = 1{,}19$ g/ml) 3,5 g of hexamethylenetetramine Distilled water to make 1 000 ml	10 min	20 °C to 25 °C	An increased number of pickling intervals or longer times may be required in certain circumstances.
C.3.2[a]		1 000 ml of hydrochloric acid (HCl, $\rho = 1{,}19$ g/ml) 20 g of antimony trioxide (Sb_2O_3) 50 g of tin(II) chloride ($SnCl_2$)	1 min to 25 min	20 °C to 25 °C	Solution should be vigorously stirred or specimen should be brushed. An increased number of pickling intervals or longer times may be required in certain circumstances.
C.3.3		500 ml of hydrochloric acid (HCl, $\rho = 1{,}19$ g/ml) 5 g of 1,3-di-*n*-butyl-2-thiourea Distilled water to make 1 000 ml	1 min to 5 min	20 °C to 25 °C	Solution should be vigorously stirred or specimen should be brushed. An increased number of pickling intervals or longer times may be required in certain circumstances.
C.4.1	Lead and lead alloys	1 500 g of ammonium acetate (CH_3COONH_4) Distilled water to make 1 000 ml (saturated solution)	5 min	20 °C to 25 °C	
C.5.1[a]	Magnesium and magnesium alloys	200 g of chromium trioxide (CrO_3) 10 g of silver nitrate ($AgNO_3$) 20 g of barium nitrate $[Ba(NO_3)_2]$ Distilled water to make 1 000 ml	1 min	20 °C to 25 °C	The silver salt is presented to precipitate chloride. The barium salt is presented to precipitate sulfate.
C.6.1	Nickel and nickel alloys	150 ml of hydrochloric acid (HCl, $\rho = 1{,}19$ g/ml) Distilled water to make 1 000 ml	1 min to 3 min	20 °C to 25 °C	
C.6.2		100 ml of sulfuric acid (H_2SO_4, $\rho = 1{,}84$ g/ml) Distilled water to make 1 000 ml	1 min to 3 min	20 °C to 25 °C	
C.7.1	Stainless steels	100 ml of nitric acid (HNO_3, $\rho = 1{,}42$ g/ml) Distilled water to make 1 000 ml	20 min	60 °C	May not be suitable for very low-alloyed stainless steels e.g. 13 % Cr steel, due to mass loss of the base metal.
C.7.2		200 ml of nitric acid (HNO_3, $\rho = 1{,}42$ g/ml) Distilled water to make 1 000 ml	60 min	20 °C to 25 °C	Start at room temperature. Heat if necessary to 60 °C. Suitable for most stainless steels.
C 7.3		500 ml of hydrochloric acid (HCl, $\rho = 1{,}19$ g/ml) 5 g of 1,3-di-*n*-butyl-2-thiourea Distilled water to make 1 000 ml	20 s to 30 s	20 °C to 25 °C	Suitable for most stainless steels, but gives more mass loss of the base metal than designation C.7.2. The mass loss is still moderate.
C.7.4		100 g of oxalic acid 900 ml of distilled water	120 min	20 °C to 25 °C	Start at room temperature. Heat if necessary to 40 °C. Steel grade 1,446 2 was exposed for 10 h at room temperature with no mass loss measured. May not be suitable for very low-alloyed stainless steels e.g. 13 % Cr steel, due to mass loss of the base metal.

Table A.1 (*continued*)

Designation	Material	Chemical products	Total time	Temperature	Remarks
C.7.5		100 g of oxalic acid 900 ml of distilled water	60 min	40 °C	Steel grade 1,446 2 was exposed for 1 h at 40 °C with no mass loss measured. May not be suitable for very low-alloyed stainless steels e.g. 13 % Cr steel, due to mass loss of the base metal.
C.7.6		500 ml of phosphoric acid (H_3PO_4, $\rho = 1,70$ g/ml) Distilled water to make 1 000 ml	60 min	20 °C to 25 °C	May not be suitable for very low-alloyed stainless steels e.g. 13 % Cr steel, due to mass loss of the base metal.
C.7.7		150 g of diammonium citrate [$(NH_4)_2HC_6H_5O_7$] Distilled water to make 1 000 ml	10 min to 60 min	70 °C	
C.7.8[b]		100 ml of nitric acid (HNO_3, $\rho = 1,42$ g/ml) 20 ml of hydrofluoric acid [HF, $\rho = 1,155$ g/ml (47 % HF to 53 % HF)] Distilled water to make 1 000 ml	1 min to 20 min	20 °C to 25 °C	This solution may effect on the mass of the base metal, especially of low-alloyed stainless steels. Use plastic equipment as glass equipment corrodes in hydrofluoric acid solutions.
C.7.9		100 g of citric acid ($C_6H_8O_7$) 50 ml of sulfuric acid (H_2SO_4, $\rho = 1,84$ g/ml) 2 g of inhibitor (di-*ortho*-tolyl thiourea or quinoline ethyl iodide or β-naphtholquinoline) Distilled water to make 1 000 ml	5 min	60 °C	
C.8.1	Tin and tin alloys	150 g of trisodium phosphate dodecahydrate ($Na_3PO_4 \cdot 12H_2O$) Distilled water to make 1 000 ml	10 min	Boiling	
C.8.2		50 ml of hydrochloric acid (HCl, $\rho = 1,19$ g/ml) Distilled water to make 1 000 ml	10 min	20 °C	
C.9.1	Zinc and zinc alloys	250 g of glycine (NH_2CH_2COOH) Distilled water to make 1 000 ml (saturated solution)	1 min to 10 min	20 °C to 25 °C	
C.9.2		100 g of ammonium chloride (NH_4Cl) Distilled water to make 1 000 ml	2 min to 5 min	70 °C	
C.9.3[a]		200 g of chromium trioxide (CrO_3) Distilled water to make 1 000 ml	1 min	80 °C	For chloride-contaminated specimens, 10 g of silver nitrate ($AgNO_3$) per 1 000 ml solution should be added to prevent hydrochloric acid attack on the base metal.
C.9.4		100 g of ammonium acetate (CH_3COONH_4) Distilled water to make 1 000 ml	2 min to 5 min	70 °C	

[a] The pickling solution has negative environmental effects and should be handled with caution.

[b] **WARNING — Care must be taken when handling hydrofluoric acid (HF), as this acid will cause severe damage if it comes into contact with the skin or eyes. Read the safety sheets**.

Table A.2 — Electrolytic cleaning procedures for removal of corrosion products

Designation	Material	Chemical products	Total time	Temperature	Remarks
E.1.1	Iron, cast iron, steel	75 g of sodium hydroxide (NaOH) 25 g of sodium sulfate (Na_2SO_4) 75 g of sodium carbonate (Na_2CO_3) Distilled water to make 1 000 ml	20 min to 30 min	20 °C to 25 °C	Cathodic treatment with 100 A/m^2 to 200 A/m^2 current density. Use a carbon, platinum or stainless steel anode.
E.1.2		28 ml of sulfuric acid (H_2SO_4, $\rho = 1,84$ g/ml) 0,5 g of inhibitor (di-*ortho*-tolyl thiourea or quinoline ethyliodide or β-naphtholquinoline) Distilled water to make 1 000 ml	3 min	75 °C	Cathodic treatment with 2 000 A/m^2 current density. Use carbon, platinum or lead anode.
E.1.3		100 g of diammonium citrate [$(NH_4)_2HC_6H_5O_7$] Distilled water to make 1 000 ml	5 min	20 °C to 25 °C	Cathodic treatment with 100 A/m^2 current density. Use a carbon or platinum anode.
E.2.1	Lead and lead alloys	28 ml of sulfuric acid (H_2SO_4, $\rho = 1,84$ g/ml) 0,5 g of inhibitor (di-*ortho*-tolyl thiourea or quinoline ethyl iodide or β-naphtholquinoline) Distilled water to make 1 000 ml	3 min	75 °C	Cathodic treatment with 2 000 A/m^2 current density. Use a carbon, platinum or lead anode.
E.3.1	Copper and copper alloys	7,5 g of potassium chloride (KCl) Distilled water to make 1 000 ml	1 min to 3 min	20 °C to 25 °C	Cathodic treatment with 100 A/m^2 current density. Use carbon or platinum anode.
E.4.1	Zinc and cadmium	50 g of of sodium hydrogenphosphate (Na_2HPO_4) Distilled water to make 1 000 ml	5 min	70 °C	Cathodic treatment with 110 A/m^2 current density. Specimen shall be energized prior to immersion. Use carbon, platinum or stainless steel anode.
E.4.2		100 g of sodium hydroxide (NaOH) Distilled water to make 1 000 ml	1 min to 2 min	20 °C to 25 °C	Cathodic treatment with 100 A/m^2 current density. Specimen shall be energized prior to immersion. Use carbon, platinum or stainless steel anode.

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