INTERNATIONAL STANDARD

ISO 8407

Third edition
2021-01

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

Reference number
ISO 8407:2021(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 262, *Metallic and other inorganic coatings, including for corrosion protection and corrosion testing of metals and alloys*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 8407:2009), which has been technically revised. The main changes compared with the previous edition are as follows:

— Table A.1 has been revised to include current chemical cleaning procedures for the removal of corrosion products.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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

WARNING — Safety rules for personnel: handling of the solutions used for the removal of corrosion products shall be left to skilled personnel or conducted under their control. The equipment shall 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 document 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 document, 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.

In some cases, these procedures are also applicable to metal coatings, providing the possible effects from the substrate are considered.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Procedures

4.1 General

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

4.1.2 If the treatment described in 4.1.1 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 can accumulate in pits.

4.1.3 An ideal procedure should remove corrosion products and not result in the removal of any base metal. Two procedures can be used to confirm this point. One procedure uses a control specimen (see 4.1.4) and the other requires a certain number of cleaning cycles on the corroded specimen (see 4.1.5). The procedures shall be maintained while the rust removal performance of the solutions listed in Tables A.1 and A.2 is not impaired.

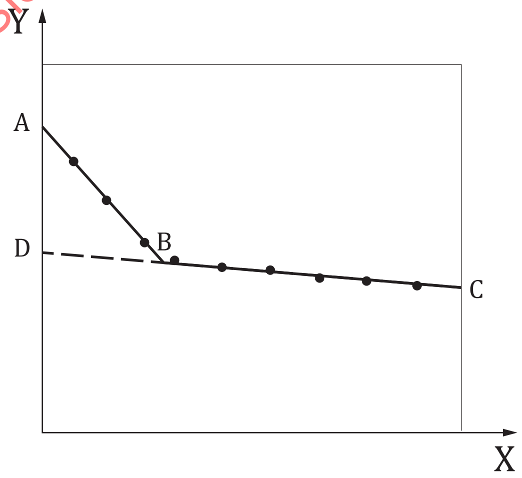
NOTE 1 Indications that the solution needs to be discarded can be discoloration or the presence of a significant amount of corrosion products in the solution.

NOTE 2 Some solutions might need some aging before working without etching the substrate.

4.1.4 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.

4.1.5 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 periods of cleaning cycles with the same interval time or time units (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 the substrate after the corrosion products are completely removed. 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 relationship may not be linear, and the most appropriate extrapolation shall then be made. If the relationship of B and C is not linear, a linear regression method, e.g. by applying the least squares from later points of cleaning cycles, can be used for estimation of the point for the D value.

If the durations of each cleaning step are not equal, the x-axis should be expressed in units of cumulative exposure time in the cleaning solution, rather than the number of cleaning steps used up to that point.



Key

X numbers of cleaning cycles or time units

Y mass

Figure 1 — Mass of corroded specimens after repetitive cleaning cycles

NOTE The number of points needed to establish the line BC is sometimes less than indicated in the figure for the specific case, especially if there is significant experience for the pickled material and solution.

4.1.6 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 provided by the corrosion products during the cleaning procedure.

4.1.7 The preferred cleaning method will be one that:

a) provides efficient removal of corrosion products;

b) provides low or zero mass loss when applied to new uncorroded specimens (see 4.1.4);

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 4.1.5).

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

4.1.9 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 in air. Drying in an air stream is strongly recommended, or optionally a hot air blower or an oven may be used. After drying, the specimens shall be allowed to cool in a dessicator to the balance room temperature before weighing.

4.2 Chemical procedures

4.2.1 Chemical procedures involve the immersion of the corroded test specimen in a chemical solution that 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, an ultrasonic bath treatment should be used.

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

4.2.3 Before the chemical treatment, clean the specimens as described in 4.1.1. In connection with the intermittent removal of specimens for weighing, brush the specimens, if necessary, to facilitate the removal of tightly adherent corrosion products.

4.3 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, an ultrasonic bath treatment should be used.

Brushing should follow electrolytic cleaning to remove any loose deposits. This will help to minimize any redeposition of metal from reducible corrosion products in solution that would reduce the measured mass loss.

4.4 Mechanical procedures

Mechanical procedures can include scraping, scrubbing, brushing, ultrasonic methods, mechanical shock and impact blasting, e.g. grit blasting, water-jet blasting. 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 only be used 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 force used in cleaning shall be maintained as constant as possible.

5 Test report

The test report shall include at least the following information:

a) a reference to this document, i.e. ISO 8407:2021;

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 the number of equal cleaning cycles;

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

e) for mechanical procedures, the specific mechanical method employed (e.g. bristle-brush scrubbing, wooden scraper), the 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 cleaning of control specimens (see 4.1.4) or from repetitive cleaning cycles (see 4.1.5) designed to assess mass loss from the removal of base metal during the cleaning process;

h) the mass loss due to corrosion (see 4.1.6);

i) any unusual features observed;

j) the date of the test.

Annex A

(informative)

Chemical and electrolytic cleaning procedures for removal of corrosion products

A.1 General

In the development of this document, 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 the 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 the preferred order. This document should be consulted for guidance in the proper application of the procedures in Tables A.1 and A.2.

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, or if the specimens are significantly corroded, 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, ρ = 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, ρ = 1,69 g/ml) 20 g of chromium trioxide (CrO_3) Add the above chemicals to distilled water to make 1 000 ml	5 min to 10 min	80 °C to boiling	Boil gently. 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 sulfonic acid Add the above chemical to distilled water to make 1 000 ml	5 min to 10 min	20 °C to 25 °C	
C.2.2[a]		500ml of hydrochloric acid (HCl, ρ = 1,19 g/ml) Add the above chemical to distilled water to make 1 000 ml	5 min to 10 min	20 °C to 25 °C	Deaerate solution with inert gas. Brushing of test specimens to remove corrosion products followed by reimmersion for 3 s to 4 s is recommended.
C.2.3		54 ml of sulfuric acid (H_2SO_4, ρ = 1,84 g/ml) Add the above chemical to distilled water to make 1 000 ml	1 min to 10 min	20 °C to 25 °C	Deaerate solution with inert gas. Brushing of test specimens to remove corrosion products followed by reimmersion for 3 s to 4 s is recommended.
C.2.4		54 ml of sulfuric acid (H_2SO_4, ρ = 1,84 g/ml) 44 ml of hydrogen peroxide (H_2O_2, ρ = 1,13 g/ml) Add the above chemicals to distilled water to make 1 000 ml	30 s to 10 min	20 °C to 25 °C	Dissolution of corrosion products is accelerated by hydrogen peroxide. This solution should be suitable for removing thick corrosion compounds.
C.3.1	Iron and steel	500 ml of hydrochloric acid (HCl, ρ = 1,19 g/ml) 3,5 g of hexamethylenetetramine Add the above chemicals to distilled water to make 1 000 ml	1 min to 25 min	20 °C to 25 °C	Mechanical cleaning treatment should first be applied to remove thicker corrosion products to reduce the picking time. Solution should be stirred with ultrasonic agitation. An increased number of pickling intervals or longer time may be required in certain circumstances. Organic inhibitors such as an Amine-based compound should be added for the thicker corrosion products because Fe(III) ions that form during pickling work as an oxidizing agent.
C.3.2[a]		1 000 ml of hydrochloric acid (HCl, ρ = 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, ρ = 1,19 g/ml) 5 g of 1,3-di-n-butyl-2-thiourea Add the above chemicals to 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.3.4[b]		200 g of sodium hydroxide ($NaOH$) 20 g granulated zinc or zinc chips Add NaOH to distilled water to make 1 000 ml	30 min to 40 min	80 °C to 90 °C	Caution should be exercised in the use of any zinc dust since spontaneous ignition upon exposure to air can occur. This method is useful to remove corrosion products in the presence of organic product such as oil or paint.
C.3.5		200 g of diammonium hydrogen citrate $[(NH_4)_2HC_6H_5O_7]$ Add the above chemical to distilled water to make 1 000 ml	10 min	23 °C	This method is specified in ISO 9227 and is useful when it is not possible to use an acidic solution.

[a] WARNING — Care shall be taken when handling hexavalent chromium (Cr(VI)) as this acid has negative environmental effects and will cause severe damage if it comes into contact with the skin or the mucous membrane. Read the safety sheets.

[b] WARNING — Care shall be taken when handling sodium hydroxide (NaOH) as this hot alkaline will cause severe damage if it comes into contact with the skin or eyes. Read the safety sheets.

[c] WARNING — Care shall 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.1 *(continued)*

Designation	Material	Chemical products	Total time	Temperature	Remarks
C.3.6		200 g of diammonium hydrogen citrate [$(NH_4)_2HC_6H_5O_7$] Add the above chemical to distilled water to make 1 000 ml	20 min	75 °C to 90 °C	This method should be suitable for removing thick corrosion compounds.
C.4.1	Lead and lead alloys	1 500 g of ammonium acetate (CH_3COONH_4) Add the above chemical to distilled water to make 1 000 ml	5 min	20 °C to 25 °C	Not efficient against sulfide corrosion products.
C.4.2		1 % mass fraction hydrochloric acid	1 min to 10 min	20 °C to 25 °C	Suitable even for removing sulfide corrosion products.
C.4.3		10 % mass fraction hydrochloric acid.	1 min to 10 min	20°C to 25 °C	Suitable even for removing sulfide corrosion products, however higher corrosion rate may occur for clean lead.
C.5.1[a]	Magnesium and magnesium alloys	200 g of chromium trioxide (CrO_3) Add the above chemical to distilled water to make 1 000 ml	1 min	20 °C to 25 °C	20 g of barium nitrate [$Ba(NO_3)_2$] should be added to precipitate sulfate. 10 g of silver nitrate ($AgNO_3$) may be added to precipitate chloride. Add the above chemicals to distilled water to make 1 000 ml.
C.5.2[a]		100 g of chromium trioxide (CrO_3) Add the above chemical to distilled water to make 1 000 ml	1 min	Boiling	10 g of silver chromate (Ag_2CrO_4) may be added to precipitate chloride. Add the above chemical to distilled water to make 1 000 ml. Boil gently.
C.6.1	Nickel and nickel alloys	150 ml of hydrochloric acid (HCl, ρ = 1,19 g/ml) Add the above chemical to 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, ρ = 1,84 g/ml) Add the above chemical to distilled water to make 1 000 ml	1 min to 3 min	20 °C to 25 °C	
C.7.1		100 ml of nitric acid (HNO_3, ρ = 1,42 g/ml) Add the above chemical to distilled water to make 1 000 ml	20 min	60 °C	May not be suitable for low Cr stainless steels, e.g. 13 % mass fraction Cr steel, due to mass loss of the base metal.
C.7.2		200 ml of nitric acid (HNO_3, ρ = 1,42 g/ml) Add the above chemical to 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, ρ = 1,19 g/ml) 5 g of 1,3-di-n-butyl-2-thiourea Add the above chemical to 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 Add the above chemical to 900 ml of distilled water	120 min	20 °C to 25 °C	Start at room temperature. Heat, if necessary, to 40 °C. Steel grade 1.4462 was exposed for 10 h at room temperature with no mass loss measured. May not be suitable for low Cr stainless steels, e.g. 13 % mass fraction Cr steel, due to mass loss of the base metal.
C.7.5		100 g of oxalic acid Add the above chemical to 900 ml of distilled water	60 min	40 °C	Steel grade 1.4462 was exposed for 1 h at 40 °C with no mass loss measured. May not be suitable for low Cr stainless steels, e.g. 13 % mass fraction Cr steel, due to mass loss of the base metal.

[a] WARNING — Care shall be taken when handling hexavalent chromium (Cr(VI)) as this acid has negative environmental effects and will cause severe damage if it comes into contact with the skin or the mucous membrane. Read the safety sheets.

[b] WARNING — Care shall be taken when handling sodium hydroxide (NaOH) as this hot alkaline will cause severe damage if it comes into contact with the skin or eyes. Read the safety sheets.

[c] WARNING — Care shall 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.1 *(continued)*

Designation	Material	Chemical products	Total time	Temperature	Remarks
C.7.6	Stainless steels	500 ml of phosphoric acid (H_3PO_4, ρ = 1,70 g/ml) Add the above chemical to distilled water to make 1 000 ml	60 min	20 °C to 25 °C	May not be suitable for low Cr stainless steels, e.g. 13 % mass fraction Cr steel, due to mass loss of the base metal.
C.7.7		150 g of diammonium hydrogen citrate [$(NH_4)_2HC_6H_5O_7$] Add the above chemical to distilled water to make 1 000 ml	10 min to 60 min	70 °C to boiling	Control of temperature in the solution is easier in boiling. Boil gently.
C.7.8		150 g of ammonium dihydrogen citrate [$(NH_4)H_2C_6H_5O_7$] Add the above chemical to distilled water to make 1 000 ml	10 min to 60 min	Boiling	Boil gently.
C.7.9[c]		100 ml of nitric acid (HNO_3, ρ = 1,42 g/ml) 20 ml of hydrofluoric acid [HF, ρ = 1,155 g/ml (47 % mass fraction HF to 53 % mass fraction HF)] Add the above chemicals to 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 Cr stainless steels, e.g. 13 % mass fraction Cr stainless steel. Use plastic equipment as glass equipment corrodes in hydrofluoric acid solutions.
C.8.1	Tin and tin alloys	150 g of trisodium phosphates dodecahydrate ($Na_3PO_4 \cdot 12H_2O$) Add the above chemical to distilled water to make 1 000 ml	10 min	Boiling	Boil gently.
C.8.2		50 ml of hydrochloric acid (HCl, ρ = 1,19 g/ml) Add the above chemical to 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) Add the above chemical to distilled water to make 1 000 ml	1 min to 10 min	20 °C to 25 °C	Solution is only used for zinc and zinc coating.
C.9.2[a]		200 g of chromium trioxide (CrO_3) Add the above chemical to distilled water to make 1 000 ml	1 min to 60 min	RT to 80 °C	Solution is suitable for zinc, pure zinc coating and other zinc coating alloyed with magnesium, aluminium or iron. For chloride-contaminated specimens, 10 g of silver nitrate ($AgNO_3$) per 1 000 ml solution or prior cleaning with ammonium chromate (20 g ammonium dichromate in 500 ml distilled water added with 500 ml aqueous ammonia) should be added to prevent hydrochloric acid attack on the base metal.
C.9.3[a]		20 g of diammonium dichromate[$(NH_4)_2Cr_2O_7$)] Add the above chemical to distilled water to make 490 ml Add 490 g of 25 % to 30 mass fraction ammonia water (ammonium hydroxide: NH_4OH) to the above solution.	15 min	20 °C to 25 °C	This solution is effective for removing a small amount of the corrosion products. If mass loss is smaller than the resolution of the balances, the solution after removing the corrosion product can be quantitatively analysed by inductively coupled plasma (ICP) and atomic absorption (AA) analyses to determine the mass loss of specimens. This method should not be used for zinc coatings alloyed with magnesium and/or aluminium.

[a] WARNING — Care shall be taken when handling hexavalent chromium (Cr(VI)) as this acid has negative environmental effects and will cause severe damage if it comes into contact with the skin or the mucous membrane. Read the safety sheets.

[b] WARNING — Care shall be taken when handling sodium hydroxide (NaOH) as this hot alkaline will cause severe damage if it comes into contact with the skin or eyes. Read the safety sheets.

[c] WARNING — Care shall 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.