SLOVENSKI STANDARD
SIST ISO 8407:1999

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Korozija kovin in zlitin - Odstranjevanje korozijskih produktov s preskušancev

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

Métaux et alliages -- Élimination des produits de corrosion sur les éprouvettes d'essai de corrosion

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

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

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

Annex A of this International Standard is for information only.

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

1 Scope

1.1 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 metals and alloys.

1.2 The procedures specified 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.

1.3 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. In some cases, this treatment will be sufficient to remove all corrosion products and no further treatment will be necessary.

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.

Whichever method is used, it may be necessary to repeat the cleaning treatment to ensure complete removal of 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 when corrosion products may accumulate in pits.

2.1.3 An ideal procedure should remove only 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 the 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 procedure should be repeated several times with the corroded test specimen after removal of corrosion products is completed. The mass can be shown on a graph as a function of the number of equal cleaning cycles (see figure 1). In many cases the masses obtained in these cleaning cycles (applied after removal of corrosion products) will have a linear relation with cleaning cycles. Two 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. By extrapolation of line BC to the ordinate axis, we obtain point D which characterizes the mass at zero number of cleaning cycles. In other cases the relation may not be linear and the most appropriate extrapolation shall then be made.

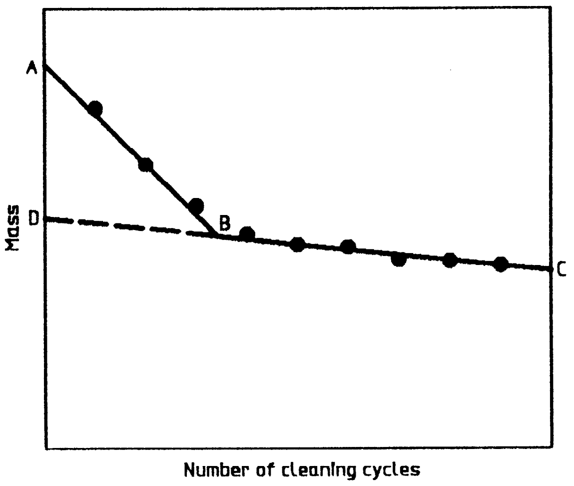

Figure 1 — Mass of corroded specimens after repetitive cleaning cycles

2.1.3.3 The true mass of the specimen after removal of the corrosion products will be between 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);

c) provides a curve of mass as a function of the number of cleaning cycles, 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 metal specimen should be thoroughly rinsed, first with tap water and finally with distilled water. Then the specimen shall be dried in a oven and allowed to cool in a desiccator to the balance room temperature before weighing. An alternative drying procedure is to immerse the metal specimen in an ethanol solution and then dry with an air hand blower.

2.2 Chemical procedure

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).

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

2.2.2 Intermittent removal of specimens from the chemical solution for light brushing can often facilitate the removal of tightly adherent corrosion products.

2.2.3 Chemical cleaning is often followed by light brushing to remove loose products.

2.3 Electrolytic procedures

Electrolytic cleaning also can be used to remove corrosion products. Several methods of electrolytic cleaning of corrosion test specimens are given in annex A (see table A.2).

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 nearly constant as possible.

3 Test report

The test report shall include the following information:

a) reference to this International Standard;

b) the procedure used to remove corrosion products;

c) for chemical procedures, the composition and concentration of chemicals employed, the solution temperature and the duration of cleaning;

d) for electrolytic procedures, the composition and concentration of chemicals employed, 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.), any 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 steps (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.

A.2 Procedures

Table A.1 and table 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. This International Standard should be consulted for guidance in the proper application of the procedures in table A.1 and table A.2.

For all the cleaning procedures listed, it is suggested that surfaces to be cleaned of corrosion products be maintained vertical. This will minimize retention of any gases released during the cleaning procedure on horizontal surfaces, which in turn may alter the uniformity of the cleaning process.

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

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

Designation	Material	Chemical	Time	Temperature	Remarks
C.1.1	Aluminium and aluminium alloys	50 ml phosphoric acid (H_3PO_4, ρ = 1,69 g/ml) 20 g chromium trioxide (CrO_3) Distilled water to make 1 000 ml	5 min to 10 min	80 °C to boiling	If corrosion product films remain, then follow with the nitric acid procedure below.
C.1.2		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.2.1	Copper and copper alloys	500 ml hydrochloric acid (HCl, ρ = 1,19 g/ml) Distilled water to make 1 000 ml	1 min to 3 min	20 °C to 25 °C	De-aeration of solution with purified nitrogen will minimize base metal removal.
C.2.2		4,9 g sodium cyanide ($NaCN$) Distilled water to make 1 000 ml	1 min to 3 min	20 °C to 25 °C	Remove copper sulfide corrosion products which may not be removed by the hydrochloric acid treatment above.
C.2.3		100 ml sulfuric acid (H_2SO_4, ρ = 1,84 g/ml) Distilled water to make 1 000 ml	1 min to 3 min	20 °C to 25 °C	Before treatment, remove bulky corrosion products, to minimize copper redeposition on specimen surface.
C.2.4		120 ml sulfuric acid (H_2SO_4, ρ = 1,84 g/ml) 30 g sodium dichromate dihydrate ($Na_2Cr_2O_7 2H_2O$) Distilled water to make 1 000 ml	5 s to 10 s	20 °C to 25 °C	Removes redeposited copper resulting from sulfuric acid treatment.
C.2.5		54 ml sulfuric acid (H_2SO_4, ρ = 1,84 g/ml) Distilled water to make 1 000 ml	30 min to 60 min	40 °C to 50 °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.

Designation	Material	Chemical	Time	Temperature	Remarks
C.3.1	Iron and steel	1 000 ml hydrochloric acid (HCl , ρ = 1,19 g/ml) 20 g antimony trioxide (Sb_2O_3) 50 g 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. Longer times may be required in certain instances.
C.3.2		50 g sodium hydroxide (NaOH) 200 g granulated zinc or zinc chips 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.
C.3.3		200 g sodium hydroxide (NaOH) 20 g granulated zinc or zinc chips 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.
C.3.4		200 g diammonium citrate [$(NH_4)_2HC_6H_5O_7$] Distilled water to make 1 000 ml	20 min	75 °C to 90 °C	
C.3.5		500 ml hydrochloric acid (HCl, ρ = 1,19 g/ml) 3,5 g hexamethylenetetramine Distilled water to make 1 000 ml	10 min	20 °C to 25 °C	Longer times may be required in certain circumstances.
C.4.1	Lead and lead alloys	10 ml acetic acid (CH_3COOH) Distilled water to make 1 000 ml	5 min	Boiling	
C.4.2		50 g ammonium acetate (CH_3COONH_4) Distilled water to make 1 000 ml	10 min	60 °C to 70 °C	
C.4.3		250 g ammonium acetate (CH_3COONH_4) Distilled water to make 1 000 ml	5 min	60 °C to 70 °C	
C.5.1	Magnesium and magnesium alloys	100 g chromium trioxide (CrO_3) 10 g silver chromate (Ag_2CrO_4) Distilled water to make 1 000 ml	1 min	Boiling	The silver salt is present to precipitate chloride.
C.5.2		200 g chromium trioxide (CrO_3) 10 g silver nitrate ($AgNO_3$) 20 g barium nitrate [$Ba(NO_3)_2$] Distilled water to make 1 000 ml	1 min	20 °C to 25 °C	The barium salt is present to precipitate sulfate.
C.6.1	Nickel and nickel alloys	150 ml hydrochloric acid (HCl, ρ = 1,19 g/ml) Distilled water to make 1 000 ml	1 min to 3 min	20 °C to 25 °C	
		100 ml sulfuric acid (H_2SO_4, ρ = 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 nitric acid (HNO_3, ρ = 1,42 g/ml) Distilled water to make 1 000 ml	20 min	60 °C	
C.7.2		150 g diammonium citrate [$(NH_4)_2HC_6H_5O_7$] Distilled water to make 1 000 ml	10 min to 60 min	70 °C	
C.7.3		100 g citric acid ($C_6H_8O_7$) 50 ml sulfuric acid (H_2SO_4, ρ = 1,84 g/ml) 2 g of inhibitor (diorthotolyl thiourea or quinoline ethyliodide or β-naphtolquinoline) Distilled water to make 1 000 ml	5 min	60 °C	