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Corrosion of metals and alloys — Method for determination of the uniform corrosion rate of stainless steels and nickel based alloys in liquids

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National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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Corrosion of metals and alloys — Method for determination of the uniform corrosion rate of stainless steels and nickel based alloys in liquids

Corrosion des métaux et alliages — Méthode pour la détermination du taux de corrosion uniforme des aciers inoxydables et des alliages à base de nickel dans les liquides

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CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
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Foreword

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The committee responsible for this document is ISO/TC 156, *Corrosion of metals and alloys*.

Introduction

Stainless steel and nickel based alloys generally show good corrosion resistance but uniform corrosion can occur in acidic and alkaline solutions. The uniform corrosion rate in aqueous solutions is usually considered to be a fairly stable process as long as the corrosive environment is constant with respect to chemical composition, temperature, and flow conditions. The uniform corrosion resistance of stainless steels and nickel based alloys is thus often determined by short-term laboratory coupons immersion tests carried out under controlled conditions. However, in real applications, some variation in service conditions can occur which might cause a temporary activation of the stainless steel or nickel based alloy surface. Temperature variations, fluctuation in the access of air or other oxidants, contact to less noble materials, e.g. mild steel, or to certain cleaning agents are all factors which under certain circumstances could cause activation.

One important property to investigate is therefore the ability for the material to passivate after activation and accordingly, this method includes activation of the specimens. The corrosion rate determined by this test can be used as a basis for material selection and to estimate the lifetime of the material.

Corrosion of metals and alloys — Method for determination of the uniform corrosion rate of stainless steels and nickel based alloys in liquids

1 Scope

This International Standard specifies the measurements of the corrosion rate of uniform corrosion for stainless steels and nickel based alloys in the intended liquids and the ability for the material to passivate after activation.

This method is intended to be used for estimation of the uniform corrosion rate in liquids, under atmospheric conditions, for the chemical industry under specific environmental conditions and not as a qualification test. It can also be used to determine iso-corrosion diagrams and at which temperature the corrosion rate exceeds 0,1 mm/a.

This International Standard is not intended for excessive corrosion rates above 1 mm/a since an even higher corrosion rate indicates that the stainless steel is not suitable in the application.

This International Standard is not intended to be used for solutions containing halides, especially chlorides, since these might cause localized corrosion.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applied. For undated references, the latest edition of the referenced document applies.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

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

ISO 17864, *Corrosion of metals and alloys — Determination of the critical pitting temperature under potientiostatic control*

3 Terms and definitions

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

4 Principle

4.1 This method involves immersion of the material in the intended liquid after the required temperature has been reached. Mass measurements are carried out before and after each of three sequential periods, 24 h, 72 h, and 72 h of immersion.

Period 1: 24 h of immersion in the liquid. When exposed to acid, the passive layer will change composition during this period. In addition, contaminations on the specimens will be removed during this period. For this reason, the first period is normally not accounted for when evaluating the corrosion rate.

Period 2: 72 h of immersion in the liquid. This period determines the corrosion rate.

Period 3: 72 h of immersion in the liquid with activation of the specimens. This period determines the corrosion rate of a specimen with an active surface and the ability to repassivate in the specific liquid.

4.2 The weight, before and after each period, is measured, the mass loss during each period is calculated, and the average corrosion rate is determined for periods 2 and 3.

4.3 In the beginning of the third period, the specimens shall be activated, according to the procedure described in 8.5, to investigate whether the material has the ability to repassivate in the specific conditions.

4.4 Activation by a potentiostat or zinc shall be used, except in strongly oxidising solutions, such as nitric acids or ferrous sulfate where a hydrochloric acid solution shall be used.

4.5 The test involves immersion of specimens in a test liquid, which is in open air. This implies that the oxygen in the aerated liquid can maintain the passive layer and the result shall therefore not be compared with results obtained in deaerated liquid.

5 Equipment

5.1 **Vessel, flask, or beaker** of inert material to the test liquid.

5.2 **Specimen holder** of inert material to the test liquid shall be used.

5.3 **Temperature controller** with an accuracy of ±1 °C.

5.4 **In case of using potentiostatic activation, a potentiostat and a reference electrode is required.** The potentiostat shall be capable of controlling the electrode potential to within ±1 mV of a present value. If the liquid in the test vessel is heated, the reference electrode shall be maintained at ambient temperature external to the test vessel via a Luggin capillary probe (see ISO 17864) or electrode shall be suitable for higher temperatures and values shall be referred to as values at ambient temperature.

5.5 **Laboratory balance.** The laboratory balance shall be able to measure with accuracy of 0,000 1 g or better.

6 Test solutions

6.1 Test solutions can be laboratory prepared or process solution. If the solution is prepared in the laboratory, it shall be made from reagent-grade chemicals and high-purity water.

6.2 Precautions shall be taken according to the chemical safety data sheet of the solution.

6.3 It is necessary to be sure that the solution does not react with the vessel.

If the solution contains hydroxides (e.g. NaOH) or fluorides (e.g. HF and wet phosphoric acid, WPA), the equipment shall be made of a resistant polymer material.

6.4 If hydrochloric acid-solution is used for activation of the specimens, it shall be prepared by mixing 1 part of HCl, mass fraction 37 % by weight HCl, with 2 parts high-purity water with a conductivity less than 1 µS/cm (ISO 3696, grade 2).

7 Specimens

7.1 At least two specimens for each of the materials shall be tested in each environmental condition. Both welded and non-welded specimens can be used.

If materials of different alloy composition are tested in the same liquid, it should be verified that this does not influence the result.

7.2 The surface area of each specimen shall be at least 20 cm^2 and influence from edges surfaces shall be minimized by minimizing the area of the edges as far as possible. The specimen's dimensions shall be measured with an accuracy of 0,1 mm and the area shall be calculated. The specimen shall be orientated so the majority of the test area is representative of the surface of interest.

7.3 The edges shall be ground down to a surface finish of at least P 120 grit paper. All the other surfaces shall represent the surface of interest. The specimens shall be left in air, at a temperature higher than the dew point temperature for at least 24 h before testing, to be able to form a stable passive film.

NOTE Wet grinding is preferred but dry grinding can also be used if care is taken to avoid overheating of the surface.

7.4 If welded specimens are used, the weld shall preferably be located in the middle of the specimens. Post weld cleaning shall be selected to be representative for the application of interest.

NOTE Surface preparation such as blasting or too rough grinding surfaces might affect the result.

7.5 The specimens shall be cleaned immediately prior to immersion by degreasing, rinsing in high-purity water followed by ethanol or a similar solvent, and air drying. After degreasing, care shall be taken not to contaminate the test surface of the specimens.

8 Procedure

8.1 The specimens shall be weighed preferably at room temperature with an accuracy of 0,000 1 g or better. The weight prior to this testing shall be registered as m_i. Weighing prior to and after testing shall be conducted at the same temperature.

8.2 Examples of the experimental equipment are shown in Figures 1 and 2. In Figure 1, the equipment consists of a flask, a specimen holder, a reflux condenser, a stirrer, and a temperature controller. In Figure 2, the specimens are mounted on a specimen holder of glass and placed in the liquid. Covers of the beakers are used to avoid evaporation.

8.3 The test solutions temperature shall be constant during the test with an accuracy of ±1 °C. Stirring shall be used if corrosion products are formed in the liquid.

NOTE Slight stirring is used to avoid corrosion products on the specimens and temperature gradients in the liquid.

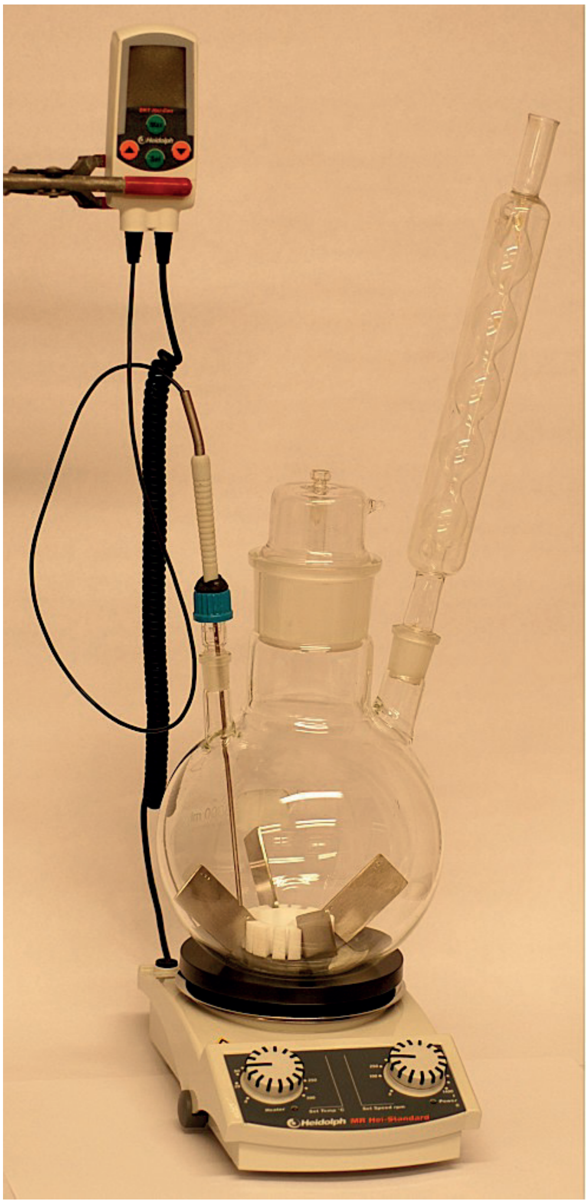

Figure 1 — Example of equipment and specimen holder

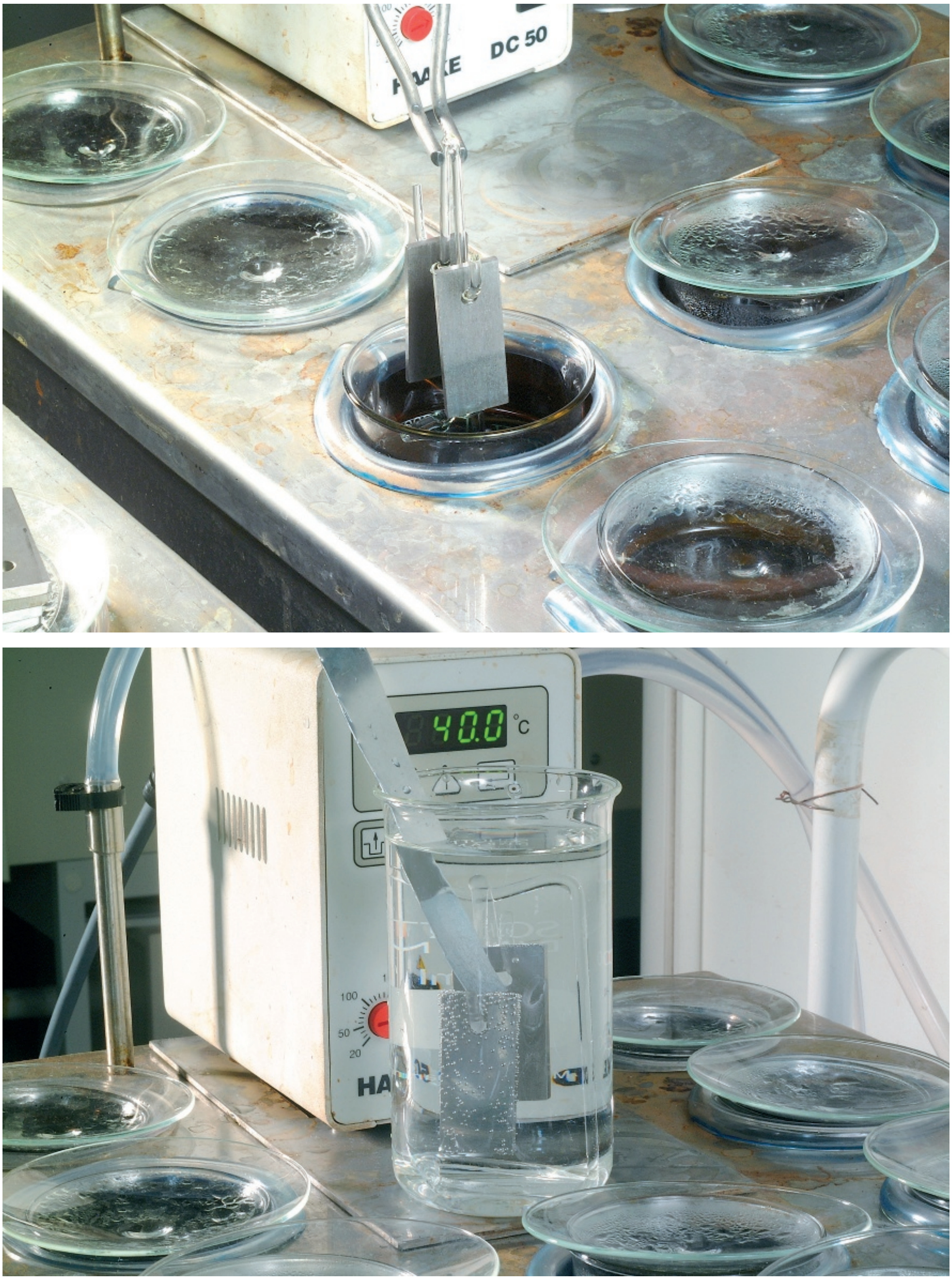

Figure 2 — Equipment consists of beakers in a thermostatic bath

8.4 The liquid is poured into the vessel, flask, or beaker. The required volume shall be at least 5 ml/cm^2 specimen area and the specimens shall be completely immersed. The temperature of the liquid shall be controlled with an accuracy of ±1 °C. In case of evaporation, a condenser shall be used to make sure that the volume is constant.

8.5 When the intended temperature has been reached and is stable, the specimens are placed in the liquid. The contact area between the specimens and the specimen holder shall be minimized. The measurements of mass loss are carried out after three sequential periods 24 h, 72 h, and 72 h.

8.6 After each test period, the specimen shall be rinsed in high-purity water (see ISO 3696). The specimens are immersed in ethanol, mass fraction 99 %, and dried in air. The weight m_{i+1} after each test period shall be noted.

NOTE i is the number of test period.

8.7 New test solutions shall be used for each period. However, if the corrosion rate for all specimens from the first period is less than 0,01 mm/a, the same solution can be used for the second period.

8.8 The specimens shall be weighed (8.1) prior to the following test period and be registered as m_i.

8.9 Activation

8.9.1 General

Before starting the last period, the specimens shall be activated. Three different kinds of activation procedures can be used; potentiostatic activation (8.9.2), zinc activation (8.9.3), or activation in HCl solution (8.9.4). For safety reasons, activation by HCl solution shall be used in strong oxidizing mineral acids or ferrous sulfate. Zinc activation is not recommended to use in alkaline solutions where pH > 8.

NOTE Corrosion test in concentrated HNO_3 does not require activation and neither in concentrated NaOH.

8.9.2 Potentiostatic activation

The specimens shall be placed in the liquid. The applied potential, E_{ap}, shall be below the activation potential. The activation potential for the specific liquid and material can be determined by obtaining a polarization curve. The E_{ap} shall be applied until gas evolution can clearly be observed.

NOTE For alloy EN 1.4652/ASTM UNS S32654 in 5 % (mass fraction) HCl at 55 °C, E_{ap} is −456 mV v.s. SHE.[3]

8.9.3 Zinc activation

The specimens shall be placed in the liquid. The zinc rod shall be in contact with the specimen by touching until gas evolution can clearly be observed on the specimen.

8.9.4 Activation in HCl solution

If the test solution contains nitric acid (HNO_3) or ferrous sulfate, the activation shall be performed in HCl solution. The solution shall be heated and the specimens shall one at a time be placed in the solution until gas evolution can clearly be observed. The specimen is then removed from the HCl solution and washed in water and high-purity water and immediately placed in the test solution. To avoid passivation of the specimen, the washing process needs to be fast.

9 Evaluation

9.1 The mass loss, $\Delta m = m_i - m_{i+1}$ of the specimen is calculated and given in g.

m_i: weight before test period i

m_{i+1}: weight after test period i

9.2 The corrosion rate is calculated according to

$$r_{\mathrm{cor}} = \frac{\mathrm{b} \cdot \Delta m}{A \cdot t \cdot \rho} \tag{1}$$

where

r_{cor}	is corrosion rate in mm/a;
b	is equal to 8760 and a conversion factor (1 year = 8 760 h);
$\Delta m = m_i - m_{i+1}$	is mass loss in g;
A	is specimen area in mm^2;
t	is exposure time in hours;
ρ	is density of the material in $\mathrm{g/mm}^3$.

NOTE 1 mm/a corresponds to 0,039 inch per year.

9.3 Calculate the corrosion rates for each period 1 to 3 and report the maximum average value from period 2 or 3 and in which period it was measured (see Annex A). All r_{cor} values above 0,1 mm/a shall be reported if the average value is below 0,1 mm/a.

NOTE During the initial time of exposure, the corrosion rate is normally higher. For this reason, the first period is commonly excluded.

9.4 In order to check for any localized corrosion, the specimens shall be examined by visual inspection and in a microscope at 20x magnification. Any localized attack shall be reported in the test report as uniform corrosion might not be the limiting corrosion mechanism.

10 Test report

The test report shall include the following information:

- a reference to this International Standard, i.e. ISO 18069:2015;
- a full description of the test material from which the specimens were taken, including composition, surface finish and structural condition, type of product, and section thickness;
- test area of specimen;
- liquid and temperature for the test;
- activation procedure;
- the maximum average corrosion rate from period 2 or 3 and in which period it was measured according to 9.2 and 9.3 (if the average value is below 0,1 mm/a, all $\mathrm{r}_{\mathrm{cor}}$ values above 0,1 mm/a shall be reported);
- localized attack, if any;
- deviations from the method described in this International Standard.

Annex A
(informative)

Example of how to calculate the corrosion rate

Table A.1 — Example of how to evaluate the measured corrosion rate

	Period 1			**Period 2**			**Period 3**		
	Specimen 1	**Specimen 2**	**Specimen 3**	**Specimen 1**	**Specimen 2**	**Specimen 3**	**Specimen 1**	**Specimen 2**	**Specimen 3**
r_{cor}, mm/a	0,213	0,356	0,274	0,091	0,015	0,013	0,138	0,156	0,184
Average corrosion rate, mm/a	0,281			0,040			0,159		

Reported corrosion rate:

0,16 mm/a in period 3.

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