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Corrosion of metals and alloys — Performance test method for corrosion inhibitors used in chemical cleaning of industry equipment

Corrosion des métaux et des alliages — Méthode d'essai de performance pour les inhibiteurs de corrosion utilisés dans le nettoyage chimique des équipements industriels

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Foreword

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This document was prepared by Technical Committee ISO/TC 156, *Corrosion of metal and alloys*.

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.

Introduction

Corrosion inhibitor can markedly decrease the corrosion rate during metals and alloys chemical cleaning process, but there is no standard for detection the performance of industry equipment chemical cleaning corrosion inhibitor. In the current environment, it is of great practical significance to establish International Standards for the performance evaluation test method of corrosion inhibitor used in industry equipment chemical cleaning process scientifically and perfectly. This document provides a basis for performance detection of corrosion inhibitor used in industry equipment chemical cleaning process and helps prevent potential hazards caused by chemical cleaning.

Corrosion of metals and alloys — Performance test method for corrosion inhibitors used in chemical cleaning of industry equipment

1 Scope

This document specifies the performance evaluation test method for corrosion inhibitors used in chemical cleaning of industry equipment. It includes instruments, reagents, preparation and pretreatment of the specimen, test conditions, test methods, reports, etc. The performance evaluation test method includes the static or the dynamic test.

This document is applicable to testing the performance of corrosion inhibitors used in chemical cleaning of industry equipment.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 6353-2, *Reagents for chemical analysis — Part 2: Specifications — First series*

ISO 7539-1, *Corrosion of metals and alloys — Stress corrosion testing — Part 1: General guidance on testing procedures*

ISO 7539-5, *Corrosion of metals and alloys — Stress corrosion testing — Part 5: Preparation and use of C-ring specimens*

ISO 11463, *Corrosion of metals and alloys — Guidelines for the evaluation of pitting corrosion*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

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

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

3.1
solubility and dispersion
ability that the corrosion inhibitor can form uniform and stable solution or form tiny particles suspended in solution without precipitation in water or cleaning liquid

3.2
time effectiveness
chemical cleaning time within which the corrosion inhibitor can maintain a good corrosion resistance performance in chemical cleaning liquid

4 Apparatus

4.1 Container used in the static test below 100 °C

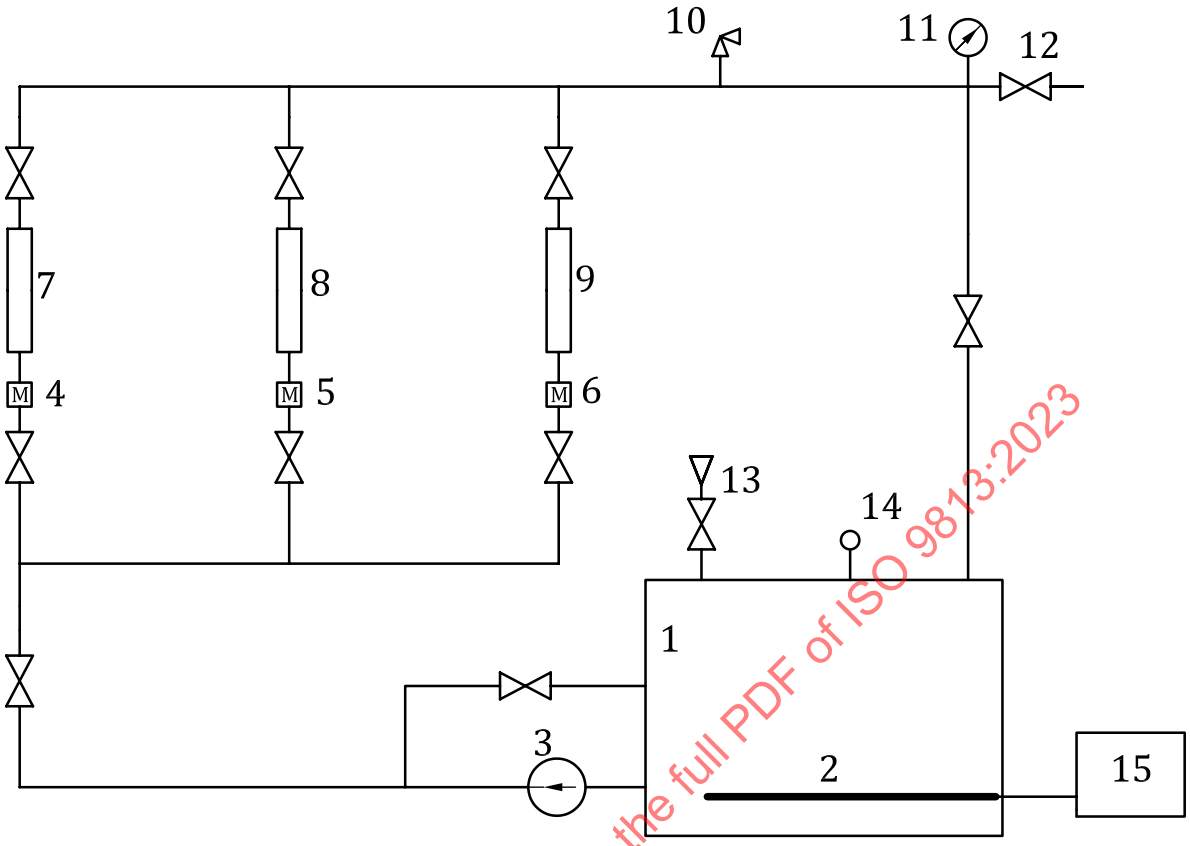
The wide-mouth jar is chosen in the static test below 100 °C. The volume of the wide-mouth jar should not be less than 500 ml. The plastic container should be used to evaluate corrosion inhibitor in hydrofluoric acid medium. A matched reflux cooler shall be equipped to the wide-mouth jar or the plastic container to prevent the solution from evaporation.

4.2 Container used in the static test above 100 °C

The autoclave is chosen in the static test above 100 °C. The volume of autoclave should not be less than 500 ml. The using temperature range of the autoclave is between 100 °C and 200 °C. The using pressure range of the autoclave is between 0,1 MPa and 2,0 MPa.

4.3 Dynamic test device

The diagrammatic sketch of dynamic test device is shown in Figure 1. The specimens can be suspended in the sample tube of dynamic test device. The flow rate in the sample tube, which the specimens are suspended to, can be adjusted and controlled. The flow rate adjustment range is from 0 m/s to 1,0 m/s and the control accuracy is ±5 %. The dynamic test device is capable of heating and controlling temperature. The temperature control accuracy is ±3 °C. If the test temperature is below 100 °C, heat resistant plastics such as polypropylene should be used in the place where the chemical cleaning liquid contact with the device. If the test temperature is above 100 °C, stainless steel should be used in the place where the chemical cleaning liquid contact with the device.



Key

1	cleaning tank	9	high speed sample tube
2	heater	10	safety valve
3	pump	11	pressure gauge
4	flowmeter	12	exhaust port
5	flowmeter	13	dosing position
6	flowmeter	14	thermometer
7	low speed sample tube	15	temperature controller
8	middle speed sample tube		

Figure 1 — Diagrammatic sketch of dynamic device

4.4 Specimen suspending device

The material used to suspend the specimen should be made of acid-resistant, heat-resistant and inert insulation material, such as synthetic fibre.

4.5 Constant temperature device

Heating sleeve, heating plate, water bath or oil bath, etc. is used to maintain the solution at the required temperature. The temperature control accuracy is ±3 °C.

4.6 Oven

The oven shall be capable of controlling temperature more than 120 °C and the precision of temperature controlling should be ±3 °C.

4.7 Analytical balance

The precision of analytical balance for weighing shall be ±0,1 mg.

4.8 Vernier caliper

The precision of vernier caliper should be ±0,02 mm.

4.9 Magnifier

The magnification of the magnifier is 10 ×.

4.10 Colorimetric tube

The volume of colorimetric tube should be 100 ml. The plastic colorimetric tube should be used to evaluate corrosion inhibitor in hydrofluoric acid medium.

4.11 Pipette

The volume of the pipette tube should be 1 ml.

5 Reagents and materials

5.1 Experimental water

The experimental water used shall conform to the specifications in ISO 3696.

5.2 Acetone

The acetone used shall conform to the specifications in ISO 6353-2.

5.3 Ethanol

The ethanol used shall conform to the specifications in ISO 6353-2.

5.4 Abrasive paper

The abrasive paper shall meet the requirements of ISO 8486-1.

6 Test specimens

The material of the specimen shall be the same as the kind of material of the system which the corrosion inhibitor is used. The dimension of the specimens should be 50 mm by 10 mm by 4 mm with two 5 mm diameter mounting hole suitably located at each end of the specimen. Each specimen shall be clearly identified.

7 Pretreatment of specimens

7.1 The specimens shall be free from oil and grease. Prior to use, the surface shall be degreased, with ethanol or acetone. A water-based degreaser can be used as long as it does not leave any residues.

7.2 All sides of specimens shall be polished with at least 120 grid abrasive paper. The grade of the final abrasive paper used should be reported. Each sheet of abrasive paper can grind only one kind of metal material.

7.3 The specimens shall be rinsed with ethanol. Then the specimens shall be rinsed with grade 3 water which conforms to ISO 3696 and dried using a hot air or an oven.

8 Test condition

8.1 Ratio of solution volume to surface area of immersed metal

The ratio of solution volume to surface area of immersed metal shall be no less than 15 ml/cm^2.

8.2 Concentration of cleaning agent

The mass concentration of cleaning agent shall be the highest concentration which is permit in the cleaning process. The water that conforms to ISO 3696 of grade 3 is recommended to dilute the cleaning agent to the certain concentration.

8.3 Concentration of corrosion inhibitor

The mass concentration of corrosion inhibitor shall be the concentration the manufacturer recommended.

8.4 Temperature

The test temperature shall be the permitted upper limit temperature of the chemical cleaning process.

8.5 Test duration

8.5.1 Corrosion rate evaluation test

The test time of corrosion rate evaluation test is 6 h.

8.5.2 Corrosion inhibition time effectiveness evaluation test

The test time of corrosion inhibition time effectiveness evaluation test is the permitted upper limit time of the chemical cleaning process.

8.6 Other provisions

If the corrosion inhibitor is utilized in static condition, the static corrosion rate evaluation test is recommended. If the corrosion inhibitor is utilized in dynamic condition, the dynamic corrosion rate evaluation test is recommended.

The performance evaluation test of corrosion inhibitor shall be conducted by the professionals and proper safety protection equipment shall be correctly worn.

9 Procedure

9.1 Static test

9.1.1 After polishing, measure the surface dimensions of the specimens to the nearest 0,02 mm. Calculate surface area of each specimen.

9.1.2 After the degreasing and drying processes, weigh the specimens to the nearest 0,1 mg. Three specimens of each kind of material are needed in the test.

9.1.3 Calculate the volume of the cleaning solution according to 8.1. Prepare the cleaning solution according to 8.2 and 8.3.

9.1.4 The specimens shall be suspended in the same level of the test solution and the specimens shall be total immersed in the solution under the depth of at least 10 mm from the solution surface. The specimens shall neither touch each other nor contact the vessel wall and the long direction of the specimen is perpendicular to the bottom of the vessel.

9.1.5 Set the test at the require temperature according to 8.4 and control the test time according to 8.5.1.

9.1.6 According to the test temperature, the following procedure is conducted respectively.

a) The static test below 100 °C.

First, add the prepared solution, the volume of which is in accordance with 9.1.3, to the test container. A matched reflux cooler is installed to the test vessel and the cooling water of the reflux cooler is opened. Second, the vessel is put into a constant temperature water bath which has reached the required test temperature. Third, when the cleaning solution is heated to the required temperature, the specimens are suspended into the test container and the reflux cooler shall be installed again. Start the test timing. When the test reaches the scheduled time, the specimens shall be taken out of the vessel.

b) The static test above 100 °C.

First, add the prepared solution, the volume of which is in accordance with 9.1.3, to the autoclave. Second, the specimens are suspended into the test autoclave and the autoclave shall be sealed. Third, start the heating device. When the cleaning solution is heated to the required temperature, start the test timing. When the test reaches the scheduled time, stop the heating device. If the temperature decreases to 70 °C or below, the specimens can be taken out of the autoclave.

9.1.7 After taking out of the specimens, the water that conforms to ISO 3696 of grade 3 is used to rinse the specimens immediately. If the specimens are not copper material, the specimens shall be washed in 1 % ammonia solution for several times. Then take out the specimens and dry the specimens using filter paper. The attachment on specimens shall be cleaned up using soft eraser. Then the specimens are immersed in the acetone and swab the surface of each specimen. After the acetone cleaning process, the specimens are dried with cool air. The surface changes are examined using a magnifier and the visible changes are recorded in Annex A. Pitting corrosion of the specimens shall be evaluated in accordance with ISO 11463. Then move the specimens into a dryer for 1 h and weigh the specimens to the nearest 0,1 mg.

9.2 Dynamic test

9.2.1 After polishing, measure the surface dimensions of the specimens to the nearest 0,02 mm. Calculate the surface area of each specimen.

9.2.2 After the degreasing and drying processes, weigh the specimens to the nearest 0,1 mg. Three specimens of each kind of material are needed in the test.

9.2.3 The volume of the cleaning solution is prepared according to the volume of the dynamic test device and the volume of the cleaning solution shall meet the requirement of 8.1. Prepare the cleaning solution according to 8.2 and 8.3.

9.2.4 Set the test at the required temperature in 8.4 and control the test time according to 8.5.1.

9.2.5 Add the prepared cleaning solution to the cleaning tank of dynamic test, start the pump to establish a self-cycle and start the heating device. When the cleaning solution is heated to the required

temperature, the specimens are suspended in the middle of the sample tube. Then establish the system cycle and adjust the flow rate of the sample tube and the specimens should be arranged in such a manner that the flow is parallel to the long direction of the specimens. After the flow rate is adjusted to the required value of the chemical cleaning process, start the test timing.

9.2.6 When the test reaches the scheduled time, stop the heating device and pump. When the temperature of the cleaning solution is dropped below 90 °C, discharge the solution in the dynamic test device and take out the specimens. Then treat the specimens according to 9.1.7.

9.3 Calculation of the corrosion rate

The corrosion rate is calculated using Formula (1).

$$r = (W_0 - W_1)/(S \times t) \quad (1)$$

where

r is the corrosion rate, expressed in grams per square metre per hour [g/(m^2·h)];

W_0 is the mass of the specimens before the test, expressed in grams (g);

W_1 is the mass of the specimens after the test, expressed in grams (g);

S is the surface area, expressed in square metres (m^2);

t is the test time, expressed in hour (h).

The result of the test is the average value of the three specimens' corrosion rate. If the relative error of the specimen is larger than 10 %, it is necessary to repeat the test. Record process information of the test and corrosion rate results in Annex A.

9.4 Intergranular corrosion and stress corrosion

If the corrosion inhibitor is utilized in the system that contain austenitic steel materials, it is necessary to conduct intergranular corrosion and stress corrosion of the specimens after the corrosion test. Intergranular corrosion shall be conducted in accordance with ISO 4214. Stress corrosion shall be conducted in accordance with ISO 7539-1 and ISO 7539-5.

9.5 Solubility and dispersion test

9.5.1 Start the constant temperature water bath and set the temperature to the required value. If the corrosion inhibitor is used in the temperature more than 100 °C, the solubility and dispersion test temperature is 95 °C.

9.5.2 Add 70 ml water that conforms to ISO 3696 of grade 3 to a colorimetric tube. Then, put the colorimetric tube into the water bath at the required temperature. When the temperature in the colorimetric tube reaches the required temperature, add 0,4 ml or the manufacture recommended amount corrosion inhibitor to the colorimetric tube. Put the plug and shake the colorimetric tube for 30 s. Put it back into the water bath. The water lever in the colorimetric tube shall be near the water level of the water bath. After 1 h, observe whether the solution precipitates.

9.5.3 Calculate the amount of cleaning agent needed in 100 ml, add the calculated cleaning agent into the colorimetric tube, constant-volume the solution to 100 ml. Put the plug and shake the colorimetric tube for 30 s. Put the solution back into the water bath at the required temperature. The water lever in the colorimetric tube shall be near the water level of the water bath. After 2 h, observe whether the solution precipitates.

9.6 Time effectiveness test

The procedure of the time effectiveness test is the same as the static test. The time effective test time is in accordance with 8.5.2.

10 Report

Report the data and process for each test performed on a form as illustrated in Annex A (see Table A.1, Table A.2 and Table A.3).

Annex A
(informative)

Result of corrosion inhibitor test

Table A.1 — Report of corrosion inhibitor corrosion rate test

Test date:

Corrosion inhibitor type			Manufacture name	
Client name			Laboratory name	
Test condition	Temperature, °C			
	Flow rate, m/s			
	Time of specimens put in			
	Time of specimens taken out			
	Test duration, h			
Concentration of cleaning agent, %				
Concentration of corrosion inhibitor, %				
Material of specimen				
Treatment of specimen surface				
Number of specimens				
Surface area of specimen, cm^2				
Mass of specimen	Mass before test, g			
	Mass after test, g			
	Mass difference, g			
Corrosion rate, $g/(m^2 \cdot h)$				
Surface change of the specimen				
Pitting corrosion				
Intergranular corrosion and stress corrosion (austenitic steel)				

Table A.2 — Report of corrosion inhibition time effectiveness test

Test date:

Corrosion inhibitor type			Manufacture name	
Client name			Laboratory name	
Test condition	Temperature, °C			
	Time of specimens put in			
	Time of specimens taken out			
	Test duration, h			
Concentration of cleaning agent, %				
Concentration of corrosion inhibitor, %				
Material of specimen				
Treatment of specimen surface				
Number of specimens				
Surface area of specimen, cm^2				
Mass of specimen	Mass before test, g			
	Mass after test, g			
	Mass difference, g			
Corrosion rate, $g/(m^2 \cdot h)$				
Surface change of the specimen				
Pitting corrosion				
Intergranular corrosion and stress corrosion (austenitic steel)				