
**Corrosion of metals and alloys —
Test method for high temperature
corrosion testing of metallic materials
by application of a deposit of salt, ash,
or other substances**

*Corrosion des métaux et alliages — Méthode d'essai pour essais
de corrosion à haute température de matériaux métalliques par
application d'un dépôt de sel, de cendres ou d'autres substances*

ISO 17224:2015

<https://standards.iteh.ai/catalog/standards/sist/f4f8f241-e8d7-4f02-9373-1c6835257d5f/iso-17224-2015>



iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 17224:2015

<https://standards.iteh.ai/catalog/standards/sist/f4f8f241-e8d7-4f02-9373-1c6835257d5f/iso-17224-2015>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2015

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Test method	2
4.1 Principle	2
4.2 Reagents and materials	2
4.2.1 Test pieces	2
4.2.2 Corrosive deposit	3
4.3 Test apparatus	3
4.3.1 Design of apparatus	3
4.3.2 Temperature monitoring	4
4.3.3 Gas supply	5
4.4 Procedure	5
4.4.1 Preparation and placement of the test piece	5
4.4.2 Test environment	6
4.4.3 Heating method	6
4.4.4 Test duration	6
4.4.5 Cooling of test pieces	7
4.4.6 Discontinuous testing	7
4.5 Determination of mass change	7
4.5.1 Principle	7
4.5.2 Measurements prior to testing	7
4.5.3 Descaling prior to mass determination	7
4.5.4 Measurement of corrosion mass loss	8
5 Examination of surface and microstructure of corroded test pieces	8
6 Report	8
6.1 Matters to be described	8
6.1.1 Test material	8
6.1.2 Test piece	8
6.1.3 Testing environments	8
6.1.4 Test results	9
6.2 Supplementary note	9
Annex A (informative) Chemical and electrolytic procedures for removal of corrosion products	10
Bibliography	13

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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information \(standards.iteh.ai\)](http://Foreword - Supplementary information (standards.iteh.ai))

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

ISO 17224:2015

<https://standards.iteh.ai/catalog/standards/sist/f4f8f241-e8d7-4f02-9373-1c6835257d5f/iso-17224-2015>

Introduction

In contrast to high temperature corrosion occurring in gaseous environment covered in ISO 21608 and ISO 13573, this International Standard focuses on high temperature corrosion occurring on materials that are in direct contact with molten corrosive substances or solid corrosive substances.

The present document considers the case of a metallic material covered with a corrosive deposit that itself can remain in the solid state or melt during the high temperature exposure.

The closely-related condition involving exposure in a compacted powder is described in ISO 17248 and that involving immersion in a molten salt or other liquid in ISO 17245.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 17224:2015

<https://standards.iteh.ai/catalog/standards/sist/f4f8f241-e8d7-4f02-9373-1c6835257d5f/iso-17224-2015>

iTeh STANDARD PREVIEW **(standards.iteh.ai)**

ISO 17224:2015

<https://standards.iteh.ai/catalog/standards/sist/f4f8f241-e8d7-4f02-9373-1c6835257d5f/iso-17224-2015>

Corrosion of metals and alloys — Test method for high temperature corrosion testing of metallic materials by application of a deposit of salt, ash, or other substances

1 Scope

This International Standard specifies the method for high temperature corrosion testing of metallic materials by applying a deposit of salt, ash, and/or other substances and exposing them to high temperature in a controlled gas environment to evaluate their corrosion resistance. The deposit is applied prior to high temperature exposure by spraying, dipping, or painting.

This International Standard does not cover methods where test pieces are fully or partially embedded in corrosive substances or immersed in liquids. These methods are covered in ISO 17248 and ISO 17245, respectively.

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 applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3611, *Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics*

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

ISO 8407, *Corrosion of metals and alloys — Removal of corrosion products from corrosion test specimens*

ISO 13385-1, *Geometrical product specifications (GPS) — Dimensional measuring equipment — Part 1: Callipers; Design and metrological characteristics*

ISO 13385-2, *Geometrical product specifications (GPS) — Dimensional measuring equipment — Part 2: Calliper depth gauges; Design and metrological characteristics*

ISO 21608, *Corrosion of metals and alloys — Test method for isothermal-exposure oxidation testing under high-temperature corrosion conditions for metallic materials*

ISO 26146, *Corrosion of metals and alloys — Method for metallographic examination of samples after exposure to high-temperature corrosive environments*

ASTM E3, *Standard guide for preparation of metallographic specimens*

ASTM E220, *Standard method for calibration of thermocouples by comparison techniques*

ASTM E230, *Standard temperature-electromotive forces tables for standardized thermocouples*

ASTM E407, *Standard practice for microetching metals and alloys*

ASTM E1350, *Standard test method for testing sheathed thermocouples prior to, during and after installation*

3 Terms and definitions

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

3.1 affected layer

layer beneath the test piece surface that is affected in its composition and/or structure due to corrosion

3.2 corrosive deposit

mixture made up of salt, ash, and/or other substances that contains compounds that are expected to react with the metal at high temperature

Note 1 to entry: The corrosive deposit can be molten at test temperature.

3.3 controlled gas environment

flowing gas mixture of defined composition and flow rate which affects the behaviour of the corrosive deposit

3.4 descaling

removal of corrosion products and corrosive phases from the test piece surface before measurement of the mass of the remaining metal

4 Test method

4.1 Principle

The test shall be performed with at least two test pieces of each material to ensure reproducibility of the test results. These test pieces can be used for determination of mass change, dimensional changes, and/or for observation of the surface and/or cross section.

4.2 Reagents and materials

4.2.1 Test pieces

The test pieces shall have the form of a rectangular plate, a disc, or a cylinder with a minimum surface area of 300 mm² and a minimum thickness of 1,5 mm .

If the test pieces cannot be made according to these specifications, the shape and dimensions of the test pieces shall be in accordance with the agreement between the parties involved.

The test pieces shall be machined to remove the strata affected by cutting.

The final finishing of the surface of the test pieces shall be performed with abrasives with mean particle diameter of approximately 15 µm. This can be achieved by the use of abrasives, according to [Table 1](#).

Table 1 — Designation and mean diameter of particles of coated abrasives according to regional standards

Standard	Designation	Mean diameter µm	Region
FEPA ^a 43–1984 R 1993: Grit sizes for coated abrasives ISO 6344 Coated abrasives - Grain size analysis	P1200	15,3 ± 1,0	Europe
JIS R6001–87	#1000	15,5 ± 1,0	Japan
ANSI B74.12–92 — Specifications for the size of abrasive grain – grinding wheels, polishing, and general industrial uses	600	16,0	America

^a Federation of European producers of abrasives.

If another surface finish is required by the parties involved, the surface finish condition shall be described.

Sharp edges of test pieces might give anomalous behaviour. These shall be slightly rounded during the final stages of test piece preparation.

The dimensions of the test pieces shall be measured prior to exposure at a minimum of three positions for each dimension with a precision of $\pm 0,02$ mm by means of the measuring instruments specified in ISO 3611, ISO 13385-1 and ISO 13385-2.

After ultrasonically degreasing in isopropanol or ethanol, the test pieces shall be dried in hot air or in a desiccator.

If it is suspected that test pieces might absorb significant amounts of atmospheric contaminants such as water, it is recommended that the cleaned test pieces are stored in a desiccator prior to weighing and exposure.

The mass of the test pieces shall be determined prior to exposure. At least two measurements shall be made for each test piece. The difference between the measurements shall not exceed 0,05 mg.

4.2.2 Corrosive deposit

The type of deposit to be used in the test shall be selected in accordance with the environment for which the test is intended. It shall be prepared either by taking ash from actual equipment or by mixing chemicals of reagent grade.

The melting temperature of the deposit should be measured in advance.

The deposit load applied will depend upon the chemistry of the deposit. Typical levels employed may range from 1 mg/cm² for sprayed salt deposits to 30 mg/cm² for suspended ash deposits.

The deposit can be applied as a solution, as a suspension, or as a dry powder. As a solution the deposit may be sprayed onto the test piece. If this technique is used it is recommended that the test piece be heated, e.g. by a hot plate.

For a suspension the corrosive deposit shall be ground in a mortar. It shall then be suspended in a liquid that does not react with the deposit or substrate. The suspension shall be mixed well. The deposit can be applied for instance by dipping or painting.

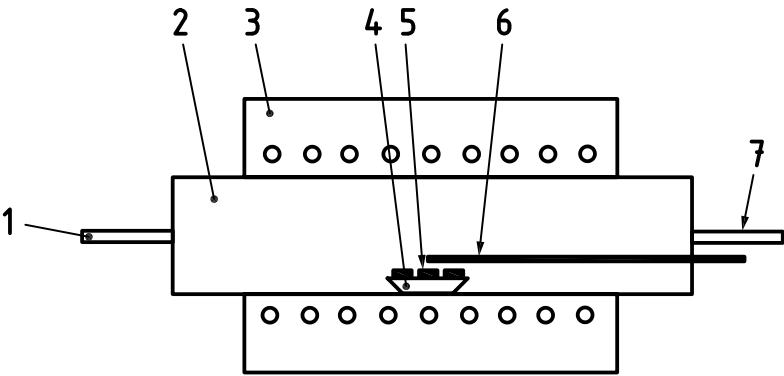
A specified amount shall be applied on the surfaces of interest. Drying is recommended prior to exposure.

4.3 Test apparatus

4.3.1 Design of apparatus

The apparatus shall be composed, as a whole, of the temperature regulating device for heating all test pieces at a uniform temperature. The heating device shall be equipped with a testing portion capable of separating the test pieces from outside air. The gas supply shall be controlled by a gas flow meter.

A basic design of a closed, horizontal, apparatus is shown in [Figure 1](#). A design of the complete furnace setup is shown in ISO 21608:2012, Figure 1.



- Key**
- 1 test gas inlet
 - 2 test chamber
 - 3 heating unit
 - 4 test piece support
 - 5 test piece
 - 6 thermocouple
 - 7 gas exit

Figure 1 — Apparatus using a horizontal furnace

The test chamber shall not be composed of materials that react significantly with the gas environment or the corrosive substance during the test. If the reaction is not avoidable, the reaction shall be small enough to neglect a change in composition of the atmosphere.

4.3.2 Temperature monitoring

The temperature distribution of the furnace shall be characterized at the exposure temperature prior to the testing to determine the width of the isothermal zone by the use of a movable thermocouple.

The temperature regulating device shall be capable to guarantee that the temperature of the test piece is kept within the permissible range given in [Table 2](#).

Table 2 — Permissible tolerance of temperature of test pieces

Temperature range, °C	≤300	300 to 600	600 to 800	800 to 1 000	1 000 to 1 200	>1 200
Temperature tolerance, °C	±2	±3	±4	±5	±7	By agreement

Thermocouple sheaths shall be used to protect the thermocouple wires. The thermocouple sheaths shall fully withstand the test temperature and environment.

A specimen thermocouple shall be positioned as close as possible to the test piece. If the experimental condition does not allow the use of such a thermocouple, the temperature of the test piece has to be deduced from the furnace calibration using dummy test pieces in an appropriate environment.

Calibration of thermocouples shall be performed in accordance with ASTM E220, ASTM E230, and ASTM E1350. A representative thermocouple taken from the batch of wire can be calibrated.

Thermocouples shall be recalibrated annually or at the beginning and the end of each experiment if there is uncertainty about thermocouple stability.

4.3.3 Gas supply

The gas supply system shall be capable of supplying the test gases at a constant flow rate to the test piece chamber described in 4.3.1.

When a humidifying regulator is used, it shall be capable of adjusting to the desired humidity. Deionized water of electrical conductivity less than $1 \mu\text{S cm}^{-1}$ shall be used.

The gas line between humidifying regulator and test chamber shall be kept above the dew point in order to avoid condensation.

The gas flow shall be monitored by a gas flow meter. The flow meter shall be located as close as possible to the inlet of the test chamber except where a humidifying regulator is used, in which case, it shall be located upstream from the humidifier.

In the case that the gas is humidified, the water vapour content shall be measured. This can be achieved by, e.g. the use of a hygrometer before the test chamber or by measuring the amount of water after condensation of the exhaust gases or by measuring the water consumption of the humidifier over the course of the experiment.

4.4 Procedure

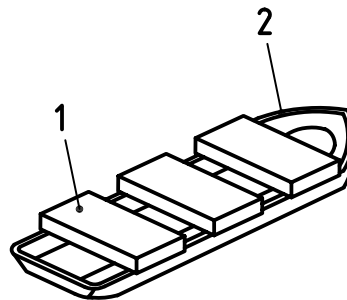
4.4.1 Preparation and placement of the test piece

The support shall be inert to the environment (e.g. alumina).

The configuration of the test piece on a support is shown in Figure 2.

Supports shall be baked in air to remove volatile compounds before their first use. The recommended baking conditions are at least 24 h at a temperature of $1\,000\text{ }^{\circ}\text{C}$. If water absorption is suspected to have occurred, used crucibles shall be dried at significantly above $100\text{ }^{\circ}\text{C}$.

In case of melting deposits, the test pieces shall be placed horizontally.



Key

- 1 test piece
- 2 inert support

Figure 2 — Test pieces on an inert support

If different materials are tested simultaneously within one furnace and if significant evaporation of the materials is expected, the test pieces shall be placed within separate horizontally oriented tubes (e.g. alumina) stacked onto each other in order to minimize cross contamination. To ensure sufficient