
**Corrosion of metals and alloys — Test
method for high temperature corrosion
testing of metallic materials by
embedding in salt, ash, or other solids**

*Corrosion des métaux et alliages — Méthode d'essai pour essais
de corrosion à haute température de matériaux métalliques par
enfouissement dans du sel, des cendres ou d'autres solides*

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Contents

	Page
Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
4 Test method	2
4.1 Principle	2
4.2 Reagents and materials	2
4.2.1 Test pieces	2
4.2.2 Corrosive powder	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	9
6.1.3 Testing environments	9
6.1.4 Test results	9
6.2 Supplementary note	10
Annex A (informative) Chemical and electrolytic procedures for removal of corrosion products from fully embedded test pieces	11
Bibliography	14

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).

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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](#).

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

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Introduction

In contrast to high temperature corrosion occurring in a gaseous environment, which is covered in ISO 21608 and ISO 13573, this International Standard focuses on high temperature corrosion occurring on materials that are in direct contact with solid corrosive substances, commonly referred to as under-deposit corrosion.

This International Standard considers the case of a metallic material embedded, partially or completely, in a corrosive powder that is present in abundance and remains completely or partially in the solid state throughout high temperature exposure. Partially embedded test pieces offer the advantage of allowing investigation of corrosion at three different positions, i.e. under the powder, at the powder/gas phase interface, and in the gas phase above the corrosive powder.

The closely-related condition involving immersion in a molten salt or other liquid is described in ISO 17245, and that involving application of a surface deposit of salt, ash, or other substances in ISO 17224.

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Corrosion of metals and alloys — Test method for high temperature corrosion testing of metallic materials by embedding in salt, ash, or other solids

1 Scope

This International Standard specifies the method for high temperature corrosion testing of metallic materials by embedding in a corrosive powder made up of salt, ash, and/or other solids and exposing them to high temperature in a controlled gas environment to evaluate their corrosion resistance.

Two options exist: fully embedding and partially embedding.

Fully embedding is used in cases in which a mass change measurement is required.

Partially embedding is used to investigate three potentially different interaction zones on a single test piece, i.e. the embedded part, the non-embedded part, and the gas/powder interface region. Therefore, gravimetric measurements are not appropriate and metallographic investigations are necessary instead.

This International Standard does not cover methods where test pieces are immersed in a liquid or cases in which a surface deposit is applied to the test pieces. These methods are covered in ISO 17245 and ISO 17224, 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 6906, *Vernier callipers reading to 0,02 mm*

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

ISO 8407:2009, *Corrosion of metals and alloys — Removal of corrosion products from corrosion test specimens*

ISO 21608:2012, *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 powder
powder mixture made up of salt, ash, and/or other solids that contains compounds that are expected to react with the metal at high temperature

3.3 controlled gas environment
flowing gas mixture of defined composition and flow rate which can affect the behaviour of the corrosive powder

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

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4.1 Principle
Mass change measurements can be performed with fully embedded test pieces. In this case, the test shall be performed with at least three test pieces of each material to ensure reproducibility of the test results. Two test pieces shall be used for determination of mass change or dimensional change, and one shall be used for observation of the surface and/or cross section.

4.2 Reagents and materials

4.2.1 Test pieces

Test pieces for fully embedding 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.

Test pieces for partially embedding shall have the form of a rectangular plate with a minimum length of 60 mm and a minimum thickness of 1,5 mm. The rectangular geometry allows accurate metallographic examinations of longitudinal sections as well as transverse sections, which is not possible with a cylindrical test piece.

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 a 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 ±0,02 mm by means of the measuring instruments specified in ISO 3611 and ISO 6906.

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 be stored in a desiccator prior to weighing and exposure.

4.2.2 Corrosive powder

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

The particle size of the powder shall not exceed 100 µm. When using reagent grade chemicals, the particle size (according to the supplier's specification) shall be recorded.

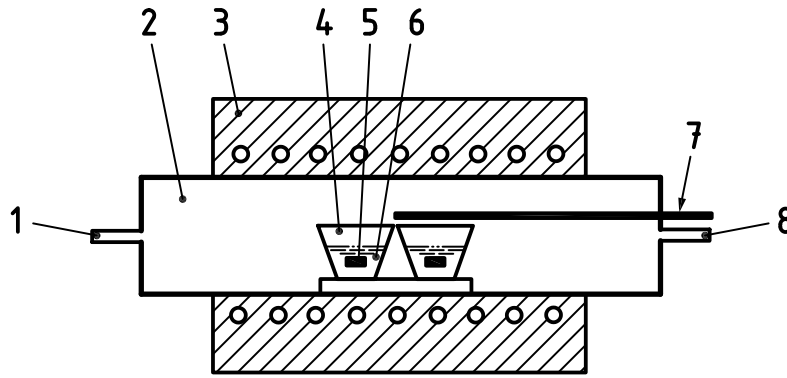
WARNING — When working with dangerous substances (such as certain ashes, molten salts, heavy metals, and organic materials), all necessary safety precautions shall be taken.

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](#). Other designs can use vertical orientation. 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 crucible
- 5 test piece
- 6 corrosive substance
- 7 thermocouple
- 8 gas exit

Figure 1 — Apparatus using a horizontal furnace

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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 of guaranteeing 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 and 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 may 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. Condensation might have a severe impact on the result.

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 crucibles shall be inert to the environment. Each crucible shall contain only one test piece.

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

For fully embedding tests, a layer of at least 3 mm of corrosive powder shall be put in the crucible and the top of the powder shall be flattened by applying a uniform pressure (about $0,04\text{ MPa} = 40\text{ g/cm}^2$).

NOTE This can be achieved by applying a flat metal piece with the appropriate mass to area ratio to the entire surface area of the powder in one or more applications.

After the test piece is set on the bed of powder, the additional powder shall be put on top of the test piece to cover it with a powder layer of at least 3 mm in thickness. Finally, the top layer shall be flattened by the procedure described above. The distance between the top of the powder and the top of the crucible shall not exceed 3 mm.

The test pieces for partially embedding tests shall be placed upright in crucibles with a height of at least the length of the test pieces and a diameter that leaves at least 5 mm on each edge of the test pieces. Corrosive powder shall be filled into the crucibles up to half the height of the test pieces and its top layer shall be flattened by the procedure described above.

The configuration of fully embedded and partially embedded test pieces in crucibles is shown in [Figure 2](#).