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Corrosion tests in artificial atmosphere at very low concentrations of polluting gas(es)

Corrosion des métaux et alliages — Essais de corrosion en atmosphère artificielle à très faible concentration en gaz polluants

Reference number
ISO 10062:2006(E)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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.

ISO 10062 was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

This second edition cancels and replaces the first edition (ISO 10062:1991), which has been technically revised.

Introduction

Products with, for example, sensitive electrical functions, may, during storage or operation in indoor locations, be affected by atmospheric corrosion dependent on climatic factors such as temperature, relative humidity, air velocity and rate of change caused by temperature and humidity. Additionally, gaseous pollutants may seriously affect the rate of corrosion, as well as the occurrence of different corrosion mechanisms. Contaminants on the surface, such as salt, dust, oil and compounds liberated from plastics, may also affect the rate and mechanism of corrosion.

Different corrosion-promoting gaseous pollutants dominate in different field environments:

- sulfur dioxide and nitrogen oxides in atmospheres influenced by combustion of fossil fuels and in traffic environments,
- hydrogen sulfide in atmospheres in the vicinity of petrochemical and steel industry, decaying organic matter, stagnant waters and animal shelters, and
- hydrogen sulfide and chlorine compounds in the vicinity of pulp and paper industry; if chlorine is used for bleaching

Those gaseous pollutants are known to act as single corrosion-promoting factors. However, in atmospheres where more than one gaseous pollutant is present, synergistic effects may be initiated. As a result, a considerable increase in the corrosion rate may occur, compared to the case when the different gaseous pollutants act as single corrosion-promoting factors.

This International Standard is intended to

a) define a general method using atmospheres polluted by one or more gases at very low volume fractions $\leqslant 10^{-6}$ under specified conditions of temperature and relative humidity, so as to avoid condensation phenomena during the test,

b) define the test apparatus and procedure required to achieve the best possible reproducibility,

c) assess performance under test conditions which accelerate corrosion; as knowledge of operating conditions proceeds, more suitable pollutants or pollutant mixtures could be used.

INTERNATIONAL STANDARD **ISO 10062:2006(E)**

Corrosion tests in artificial atmosphere at very low concentrations of polluting gas(es)

WARNING — Safety rules for personnel: Handling of the gases used for testing can be hazardous and must be left to skilled chemists or conducted under their control. The test equipment must be used and maintained by skilled personnel, not only so that the tests can be performed correctly, but also because of the hazards to health and safety that are involved.

1 Scope

This International Standard specifies tests which are intended to determine the influence of one or more flowing polluting gas(es) at volume fractions less than or equal to 10^{-6} on test samples and/or articles of metals and alloys with or without corrosion protection under determined conditions of temperature and relative humidity.

These tests apply to

a) metals and their alloys,

b) metallic coatings (anodic and cathodic),

c) metals with conversion coatings,

d) metals with anodic oxide coatings, and

e) metals with organic coatings.

2 Normative references

The following referenced documents are indispensable for the application 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 554, *Standard atmospheres for conditioning and/or testing — Specifications*

ISO 558, *Conditioning and testing — Standard atmospheres — Definitions*

ISO 7384, *Corrosion tests in artificial atmosphere — General requirements*

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

3 Terms and definitions

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

3.1
exposure time
interval of time elapsed between the moment when the specimens are introduced into the test chamber and the end of the test

3.2
test duration
interval of time during which the specimens are exposed to polluting gases

4 Apparatus

Special attention shall be paid to equipment design and the choice of construction materials, in order that the conditions (less than ± 1 °C for temperature and less than ± 3 % for relative humidity) are uniformly consistent throughout the working space, able to ensure no condensation in the test chamber, and are repeatable.

The apparatus shall enable the use, singly or in mixtures, of polluting gas(es) such as SO_2, H_2S, Cl_2 and NO_2 up to at least the concentration required, for each gas, by the test method.

The important parameters are

a) the materials used for the test chamber and the gas handling system,

b) the geometry of the test chamber,

c) the rate and pattern of the gas flow,

d) the homogeneity of gas mixing,

e) the incident illumination (see A.1.6)

The apparatus shall be constructed according to Annex A and shall meet its requirements.

A typical apparatus for polluting-gas corrosion tests is also shown in Annex B.

5 Test methods

5.1 Test severity

5.1.1 General

The test severity, to be given in the relevant specification, is defined by

- the type, concentration and flow rate of polluting gas,
- the temperature,
- the relative humidity, and
- the test duration.

5.1.2 Suggested test methods

The method to be used depends upon the purposes of the test, the nature of the metals and alloys and the type of protection to be tested.

- **Method A**

Polluting gas	SO_2 at a volume fraction of $(0{,}5 \pm 0{,}1) \times 10^{-6}$
Temperature and relative humidity	(40 ± 1) °C and (80 ± 5) % RH
	or (25 ± 1) °C and (75 ± 3) % RH

The condition of (40 ± 1) °C and (80 ± 5) % is more aggressive than the condition of (25 ± 1) °C and (75 ± 3) % RH. Either of them shall be optionally selected and be stated in the test report.

- **Method B**

Polluting gas	H_2S at a volume fraction of $(0{,}10 \pm 0{,}02) \times 10^{-6}$
Temperature and relative humidity	(40 ± 1) °C and (80 ± 5) % RH
	or (25 ± 1) °C and (75 ± 3) % RH

The condition of (40 ± 1) °C and (80 ± 5) % RH is more aggressive than the condition of (25 ± 1) °C and (75 ± 3) % RH. Either of them shall be optionally selected and be stated in the test report.

- **Method C**

Polluting gas	Cl_2 at a volume fraction of $(0{,}02 \pm 0{,}005) \times 10^{-6}$
Temperature and relative humidity	(40 ± 1) °C and (80 ± 5) % RH
	or (25 ± 1) °C and (75 ± 3) % RH

The condition of (40 ± 1) °C and (80 ± 5) % RH is more aggressive than the condition of (25 ± 1) °C and (75 ± 3) % RH. Either of them shall be optionally selected and be stated in the test report.

- **Method D**

Mixture of polluting gases	SO_2 at a volume fraction of $(0{,}5 \pm 0{,}1) \times 10^{-6}$, and
	H_2S at a volume fraction of $(0{,}10 \pm 0{,}02) \times 10^{-6}$
Temperature and relative humidity	(40 ± 1) °C and (80 ± 5) % RH
	or (25 ± 1) °C and (75 ± 3) % RH

The condition of (40 ± 1) °C and (80 ± 5) % RH is more aggressive than the condition of (25 ± 1) °C and (75 ± 3) % RH. Either of them shall be optionally selected and be stated in the test report.

- **Method E**

Mixture of polluting gases	SO_2 at a volume fraction of (0,20 ± 0,05) $\times 10^{-6}$, and NO_2 at a volume fraction of (0,5 ± 0,1) $\times 10^{-6}$
Temperature and relative humidity	(40 ± 1) °C and (80 ± 5) % RH or (25 ± 1) °C and (75 ± 3) % RH

The condition of (40 ± 1) °C and (80 ± 5) % RH is more aggressive than the condition of (25 ± 1) °C and (75 ± 3) % RH. Either of them shall be optionally selected and be stated in the test report.

- **Method F**

Mixture of polluting gases	SO_2 at a volume fraction of (0,5 ± 0,1) $\times 10^{-6}$, H_2S at a volume fraction of (0,10 ± 0,02) $\times 10^{-6}$, and Cl_2 at a volume fraction of (0,02 ± 0,005) $\times 10^{-6}$
Temperature and relative humidity	(40 ± 1) °C and (80 ± 5) % RH or (25 ± 1) °C and (75 ± 3) % RH

The condition of (40 ± 1) °C and (80 ± 5) % RH is more aggressive than the condition of (25 ± 1) °C and (75 ± 3) % RH. Either of them shall be optionally selected and be stated in the test report.

NOTE 1 Since the corrosivity is different for methods A, B, C, D, E and F the result obtained by these methods are not comparable.

NOTE 2 Other methods may be added in the future if required (e.g. increase in the relative humidity).

5.1.3 Test duration

The test duration for each test method depends upon the purposes of the test, the nature of the metals and alloys and the means of protection tested.

5.2 Treatment of the specimens prior to testing (see Clause 6)

The relevant specification may require a preconditioning of specimens, for example chemical or mechanical cleaning of the surface.

5.3 Examination of the specimens prior to testing

Measurements shall be carried out as required by the relevant specification (see ISO 7384).

5.4 Filling of the test chamber

The filling of the chamber by the specimens shall satisfy the following conditions:

- the total surface of the test specimens shall not exceed the critical level at which the concentrations of polluting gases would decrease by more than 10 % during the passage through the working space;
- the specimens shall be placed in the working space, as defined in the relevant specification.

Care shall be taken that the specimens are placed so that they do not come into contact with one another or with the walls of the chamber, and that they do not shield one another from the test atmosphere.

The specimens shall be distributed uniformly in the working space.

5.5 Procedure

Two procedures for the execution of the test may be adopted, the reference method and the simplified method.

5.5.1 Basis for calculation

5.5.1.1 Exposure time:

a) For the reference method, exposure time corresponds to t_1 to t_3 (see Figure 1).

b) For the simplified method, exposure time corresponds to t_2 to t_3 (see Figure 2).

5.5.1.2 Test duration:

a) For the reference method, test duration corresponds to t_2 to t_3 (see Figure 1).

b) For the simplified method, test duration corresponds to t_5 to t_3 (see Figure 2).

5.5.2 Reference method

The conditions of the specimens and the test chamber shall be such that condensation does not occur on the specimens when they are introduced into the test chamber.

The temperature and relative humidity inside the chamber shall be adjusted according to the specified method (see 5.1.2 and A.1.4).

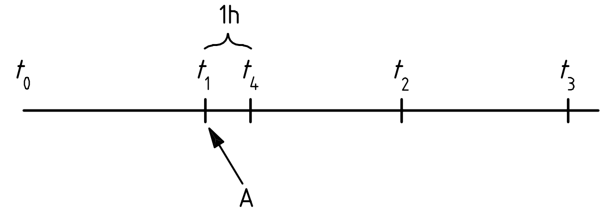
After introducing the specimens, a waiting period of 1 h should be allowed for the temperature and the relative humidity to stabilise before the polluting gas(es) is/are introduced into the test chamber.

The polluting gas(es) shall be introduced simultaneously into the damp air flow and their concentrations shall be adjusted to the value specified by the test method; during this adjustment, the maximum specified concentrations should not be exceeded (see 5.1).

The concentration of polluting gas(es) shall be stabilized in less than 1 h.

The test duration shall be measured as indicated in 5.5.1.2.

At the end of the test, the injection of polluting gas(es) shall be stopped and the specimens shall be maintained in the flow of the damp atmosphere without polluting gas(es) for 2 h.



t_0 to t_1 temperature, relative humidity adjustment

t_1 to t_4 waiting period of 1 h

t_4 to t_2 gas-concentration adjustment

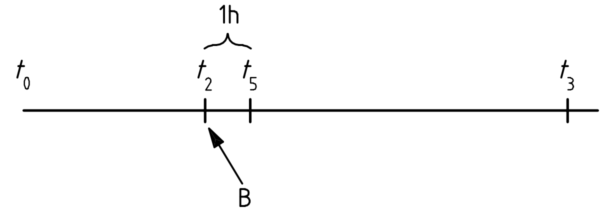
t_2 to t_3 test duration

t_1 to t_3 exposure time

A introduction of specimens

t_4 recovery of temperature

Figure 1 — Reference method



t_0 to t_2 temperature, relative humidity and gas-concentration adjustment

t_2 to t_5 waiting period of 1 h

t_5 to t_3 test duration

t_2 to t_3 exposure time

B introduction of specimens

t_5 recovery of temperature

Figure 2 — Simplified method

5.5.3 Simplified method

It is possible to stabilise the conditions of temperature, relative humidity and gas composition within the test chamber and then to introduce the test specimens quickly so that the test conditions will again be stabilised at the prescribed levels after about 1 h. If condensation on the specimens surface can then be avoided, the simplified method can be used.

The test duration shall be measured as indicated in 5.5.1.2.

At the end of the test, the injection of polluting gas(es) shall be stopped and the specimens shall be maintained in the flow of the damp atmosphere without polluting gas(es) for 2 h.

5.6 Monitoring of test behaviour

In order to monitor the chamber performance for each test, appropriate metal control coupons (copper or silver) should be exposed simultaneously with the specimens in the working space of the test chamber, and the corrosive effects on these coupons should be assessed (other examples: copper, silver, nickel, and gold on copper).

5.7 Post-test storage

The specimens shall be removed from the chamber and stored under standard conditions as specified in ISO 554 and ISO 558. The reaction of specimens during storage after testing shall be considered.

6 Information to be given in the relevant specification

The following information shall be given in the relevant specification:

a) a reference to this International Standard;

b) conditions and duration of the test;

c) treatment of the specimens prior to testing if required;

d) a report on the examination of the specimens prior to testing;

e) assessment of corrosion effects and a report on the visual examination during and after testing.

7 Expression of results

There are many criteria for the evaluation of the corrosion resistance of metals and alloys with and without corrosion protection, for example:

a) change in the specimen appearance during the test;

b) time elapsed before the first local sign of corrosion of base metal or coating appears;

c) number, depth and distribution of corrosion defects;

d) change in mass (see ISO 8407);

e) changes in dimensions (especially thickness);

f) change in mechanical, electrical, optical and other properties.

8 Test report

The test report shall contain the following information:

a) the purpose of the test;

b) a reference to this International Standard and the test method used (A, B, C, D, E or F) together with option of temperature and humidity;

c) designation and description of the test specimens and control coupons, if used (chemical composition, shape and dimensions, method of treatment: chemical, thermal, and mechanical, type or coating and its thickness);

d) known characteristics of the specimens tested;

e) exposure period;

f) duration of the test;

g) method of placing and material for fastening the test specimens during the test;

h) the results of evaluation of corrosion changes for the tested specimen surfaces, including both descriptive and numerical assessment, possibly with photographs of the test specimens;

i) comparison of control coupons with previous exposures;

j) the dates of the test.

If the necessary measurements cannot be made within the specified time, the period of storage under recovery conditions may be extended. This extension shall be mentioned in the test report.

Other data may be included in the test report depending on the purpose of the test, as well as on the criteria chosen for expressing the results.

Annex A
(informative)

Specifications for apparatus for corrosion tests in artificial atmospheres

A.1 Test chamber

A.1.1 Materials

Precaution shall be taken to compensate for

- adsorption and desorption of gases and steam,
- corrosion of the test chamber by the test atmosphere

This requires that

- the internal walls and parts of the chamber shall be made of inert material [e.g.: glass or polytetrafluoroethylene (PTFE)],
- any windows(s) shall be of PTFE or glass,
- pipes, valves and taps in contact with the polluting gas(es) should preferably be made of inert material,
- gaskets and seals shall be made of inert materials (e.g. fluoro-elastomers).

Other materials may be used, but adsorption or desorption phenomena shall be taken into account.

A.1.2 Design

The chamber shall be designed to avoid condensation at any point and to allow easy and thorough cleaning of inner walls and fittings.

The volume of the chamber shall be not less than 0,1 m^3.

In the working space, the uniformity of the temperature shall be better than ± 1 °C, and the uniformity of the relative humidity shall be better than ± 3 %.

A.1.3 Pressure seals

The test chamber shall be fitted with suitable gasket seals to allow the test conditions to be maintained and monitored.

A.1.4 Test atmosphere

The flow rate within the chamber shall be controlled. The test atmosphere shall flow through the chamber in such a manner as to ensure uniform test conditions in the working space.

This flow rate shall be sufficient to ensure at least three exchanges per hour of the atmosphere in the test chamber.

NOTE The mass transfer characteristics for the transport of pollutants to the surface of the test specimens vary with volume flow rate of test atmosphere through the test chamber, but also with the geometry of the test chamber. Because of that, deviation in corrosion rate may therefore result if two test chambers with different geometries are used, even if the exchange rate of the test atmosphere is the same in the two test chambers.

The test atmosphere is preferably injected through opening(s) in the bottom of the chamber and extracted through opening(s) in the top of the chamber. Suitable baffles shall be placed in front of the opening(s), to assure the required uniform test conditions in the working space.

The test atmosphere extracted from the chamber shall be collected in such a manner as to conform with legal safety standards.

A.1.5 Sampling points

Sampling points for the analysis of the atmosphere during the test (see A.3) are placed close to the injection points and the outlet points, in the working space. Checks should be made to ensure that the concentration of polluting gas(es) does(do) not decrease by more than 10 % during the passage through the working space.

Condensation or adsorption in the lines used to monitor the test atmosphere shall be avoided as far as possible, as this would impair the measurements.

A.1.6 Illuminance level

The test chamber shall not be exposed to direct sunlight and the level of illumination (due to daylight or to incandescent or fluorescent lamps) measured inside the test chamber at any point on the exposed surface shall not exceed 300 lx.

A.2 Generation of the test atmosphere

A.2.1 General

The test atmosphere is obtained by continuously introducing the necessary quantity of polluting gas(es) into a damp air flow to obtain the required concentration(s). The mixing of gases usually requires a mixing tube or chamber.

The dry polluting gas(es) and the damp air shall be mixed together before injection into the working space.

A.2.2 Damp-atmosphere generation

The damp atmosphere shall be within the tolerances for the specified temperature and relative humidity when it is injected into the test chamber. Water droplets or aerosols shall not be injected into the test chamber. The air used may be taken from pressurised cylinders or delivered by a dry pump after filtration and purification by activated charcoal or molecular sieve (size 5a) and a particulate filter.

A.2.3 Polluting gas(es)

The required concentrations of the polluting gas(es) can be obtained from pressurised cylinders containing mixtures of the pure polluting gas and an inert gas such as nitrogen. Permeation tubes filled with liquid or gas may also be used. The gas(es) shall be free of contaminants that may also influence the corrosion process.

A.3 Checks and control

The parameters temperature, relative humidity, gas flow and concentration of polluting gas(es) shall be maintained between the specified limits.

A.3.1 Temperature and relative humidity

Temperature and relative humidity shall be measured in the working space.

The apparatus used to measure the temperature and relative humidity shall have a response time of less than 30 s.

It is strongly recommended that these two parameters be recorded continuously.

The sensor shall be maintained in calibration because polluting gas(es) can affect it.

A.3.2 Test-gas flow rate

The overall flow rate of the gas mixture passing through the test chamber shall be kept constant by means of a control system. Continuous monitoring of the gas flow rate is necessary, using a ball-float flow meter or other suitable flow meter.

A.3.3 Concentration of polluting gas(es)

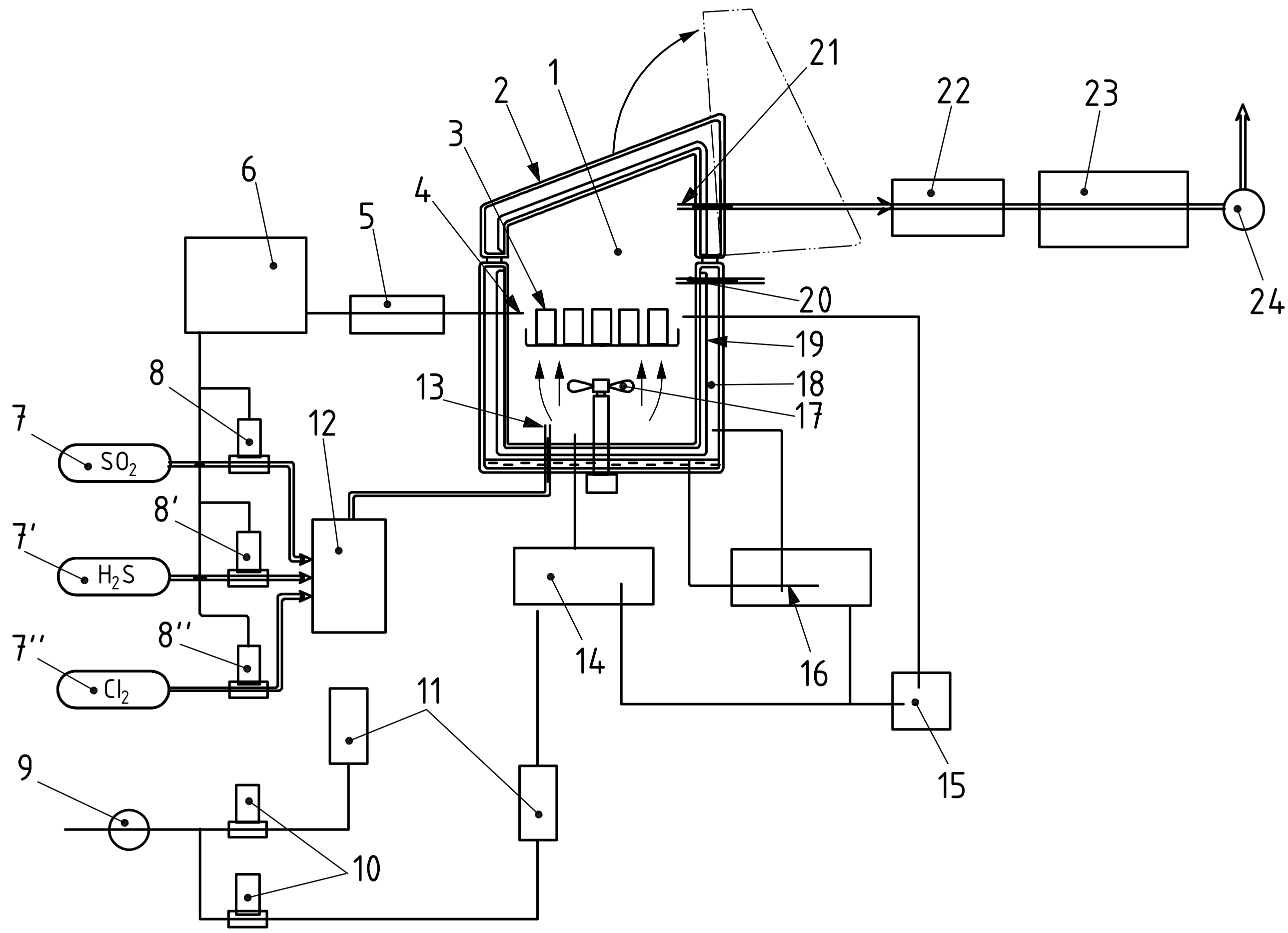
The means of analysis shall permit continuous or periodic (at least once per day) measurement of the concentration(s) of polluting gas(es), in order that these parameters may be controlled.

A.3.4 Operation

The apparatus shall allow the test to be conducted without interruption.

Annex B
(informative)

Typical apparatus for polluting gas corrosion tests



Key

1	test chamber	13	gas-injection opening
2	lid	14	humidifier
3	test specimens	15	humidity and temperature controls
4	gas-monitoring sensor	16	heating and cooling unit
5	gas analyser	17	fan
6	gas-concentration controls	18	air jacket
7	7′, 7″ gas cylinder (SO_2, H_2S, Cl_2, and/or NO_2)	19	heating and cooling system
8	8′, 8″ mass flow controller(s)	20	sampling port, manual
9	air pump	21	exhaust port
10	mass flow controls	22	dehumidifier
11	air filters	23	exhaust-gas treatment apparatus
12	air-mixing chamber	24	air-discharging blower

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