
**Metallic coatings — Test methods for
electrodeposited gold and gold alloy
coatings —**

Part 2:

**Mixed flowing gas (MFG) environmental
tests**

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*Revêtements métalliques — Méthodes d'essai des dépôts électrolytiques
d'or et d'alliage d'or —*

Partie 2: Essais climatiques aux gaz mixtes à écoulement (MFG)

<https://standards.iteh.ai/catalog/standards/sist/21a9ee1c-4299-4b37-8145-85575bcab9fd/iso-4524-2-2000>



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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 3.

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 part of ISO 4524 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 4524-2 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 7, *Corrosion tests*.

This second edition cancels and replaces the first edition (ISO 4524-2:1985), which has been technically revised.

ISO 4524 consists of the following parts, under the general title *Metallic coatings — Test methods for electrodeposited gold and gold alloy coatings*:

- Part 1: Determination of coating thickness [ISO 4524-2:2000](https://standards.iteh.ai/catalog/standards/sist/21a9ee1c-4299-4b37-8145-85575bcab9f1/iso-4524-2-2000)
- Part 2: Mixed flowing gas (MFG) environmental tests
- Part 3: Electrographic tests for porosity
- Part 4: Determination of gold content
- Part 5: Adhesion tests
- Part 6: Determination of the presence of residual salts

Annexes A, C and E form a normative part of this part of ISO 4524. Annexes B and D are for information only.

Introduction

Mixed flowing gas (MFG) tests are used to simulate or amplify exposure to environmental conditions that electrical contacts or connectors can be expected to experience in various application environments (see references [2] and [3]).

Test samples that have been exposed to MFG tests have ranged from bare metal surfaces, to electrical connectors and to complete assemblies.

The specific test conditions are usually chosen so as to simulate, in the test laboratory, the effects of certain representative field environments or environmental severity levels on standard metallic surfaces, such as copper and silver coupons or porous gold coatings (see references [2] and [3]).

Because MFG tests are simulations, both the test conditions and the degradation reactions (chemical reaction rate, composition of reaction products, etc.) may not always resemble those found in the service environment of the product being tested.

The MFG exposures are generally used in conjunction with procedures that evaluate contact or connector electrical performance such as measurement of electrical contact resistance before and after MFG exposure.

The MFG tests are useful for connector systems, the contact surfaces of which are electroplated or clad with gold or other precious metal finishes. For such surfaces, environmentally produced failures are often due to high resistances or intermittences caused by the formation of insulating contamination in the contact region. This contamination, in the form of films and hard particles, is generally the result of pore corrosion product migration or tarnish creepage from pores in the precious metal coating and from unplated metal boundaries, if present.

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Metallic coatings — Test methods for electrodeposited gold and gold alloy coatings —

Part 2: Mixed flowing gas (MFG) environmental tests

WARNING — This part of ISO 4524 does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this part of ISO 4524 to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This part of ISO 4524 provides procedures for conducting mixed flowing gas (MFG) environmental tests involving exposures to controlled quantities of corrosive gas mixtures.

It describes the required equipment and the methods for gas, temperature and humidity control that enable tests to be conducted in a reproducible manner. Reproducibility is measured through the use of control coupons the corrosion films of which are evaluated by mass gain, coulometry or by various electron and X-ray beam analysis techniques. Reproducibility can also be measured by *in situ* corrosion rate monitors using electrical resistance or mass frequency change methods.

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MFG tests can be used to:

- evaluate novel electrical contact metallization for susceptibility to degradation due to environmental exposure to the test corrosive gases;
- evaluate the shielding capability of connector housings that may act as a barrier to the ingress of corrosive gases;
- evaluate the susceptibility of other connector materials such as plastic housings to degradation from the test corrosive gases.

MFG tests are not normally:

- used as porosity tests (for guidance on porosity testing, see ISO 10308, *Metallic coatings — review of porosity tests*);
- applicable where the failure mechanism is other than pollutant gas corrosion such as in tin-coated separable contacts.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 4524. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 4524 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 4524-2:2000(E)

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 4221:1980, *Air quality — Determination of mass concentration of sulphur dioxide in ambient air — Thorin spectrophotometric method.*

ISO 4677-1:1985, *Atmospheres for conditioning and testing — Determination of relative humidity — Part 1: Aspirated psychrometer method.*

ISO 4677-2:1985, *Atmospheres for conditioning and testing — Determination of relative humidity — Part 2: Whirling psychrometer method.*

ISO 6349:1979, *Gas analysis — Preparation of calibration gas mixtures — Permeation method.*

ISO 6767:1990, *Ambient air — Determination of the mass concentration of sulfur dioxide — Tetrachloromercurate (TCM)/pararosaniline method.*

ISO 7934:1989, *Stationary source emissions — Determination of the mass of concentration of sulfur dioxide — Hydrogen peroxide/barium perchlorate/Thorin method.*

ISO 7996:1985, *Ambient air — Determination of the mass concentration of nitrogen oxides — Chemiluminescence method.*

ISO 9096:1992, *Stationary source emissions — Determination of concentration and mass flow rate of particulate material in gas-carrying ducts — Manual gravimetric method.*

ISO 10313:1993, *Ambient air — Determination of the mass concentration of ozone — Chemiluminescence method.*

ISO 12687:1996, *Metallic coatings — Porosity tests — Humid sulfur (flowers of sulfur) test.*

IEC 60050, *International Electrotechnical Vocabulary.*

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3 Terms and definitions

For the purposes of this part of ISO 4524, the terms and definitions relating to electrical contacts given in IEC 60050 apply.

4 Reagents and materials

4.1 Water for humidity generation, equivalent to grade 1 or better of ISO 3696.

4.2 Carrier gas, such as nitrogen. It shall not introduce reactive constituents into the test atmosphere to an amount of more than 5 % of any specified corrosive test atmosphere constituent.

4.3 Clean filtered air, as required for make-up to support the necessary exchange rate, in accordance with 6.6.7.1 b), as specified in 5.3.2.

4.4 Corrosive gases, chemically pure grade or equivalent.

4.5 Corrosivity monitor materials (CMMs), comprised of the coupons that are exposed to the test atmosphere for mass gain (annex A), coulometric reduction in accordance with annex C, or resistance monitoring in accordance with annex E.

5 Apparatus

5.1 General

Apparatus required to conduct MFG tests is divided into four major categories: corrosion test chamber, gas supply system, chamber monitoring system and chamber operating system.

5.2 Corrosion test chamber

5.2.1 The chamber shall consist of an enclosure made of non-reactive, low-absorbing, non-metallic materials contained within a cabinet or oven capable of maintaining the temperature to a maximum tolerance of $\pm 1^\circ\text{C}$ with a preferred tolerance held to $\pm 0,1^\circ\text{C}$ within the usable chamber working space in accordance with 6.3 with a means of introducing an exhaust gas from the chamber. The chamber isolates the reactive gases from the external environment. Chamber materials that are not low-absorbing can affect test conditions by absorbing or emitting reactive gases, leading to control and reproducibility problems. The chamber construction shall be such that the leak rate is less than 3 % of the volume exchange rate.

5.2.2 The chamber shall have provision for maintaining uniformity of the average gas flow velocity within $\pm 20\%$ of the specified value or of the chamber average when the chamber is empty. For chambers with a dimension of more than 0,1 m, measurement points shall be in accordance with A.3.2. For chambers with all dimensions of less than 0,5 m, a minimum of five points shall be measured at locations in the plane of sample exposure (perpendicular to the expected flow direction) that are equidistant from each other and the walls of the chamber. After all five or more data values are recorded, all the measurements shall be repeated a second time. When the two sets of measurements have been recorded a third complete set shall be recorded. The arithmetic average of the 15 or more measurements shall be the chamber average, see 6.5 and 6.6.8. If a hot wire anemometer is used for velocity measurements these shall be made in accordance with ISO 9096, with the exception that sample sites shall be in accordance with annex A.

5.2.3 A sample access port is desirable. This should be designed such that control coupons can be removed or replaced without interrupting the flow of gases. Corrosion test chamber corrosion rates have been shown to be a function of the presence or absence of light (see [4] and [5]). Provision shall be made for controlling the test illumination in accordance with a test specification.

5.2.4 Examples of test chamber systems are shown in Figures 1, 2 and 3.

They shall not be considered as exclusive examples.

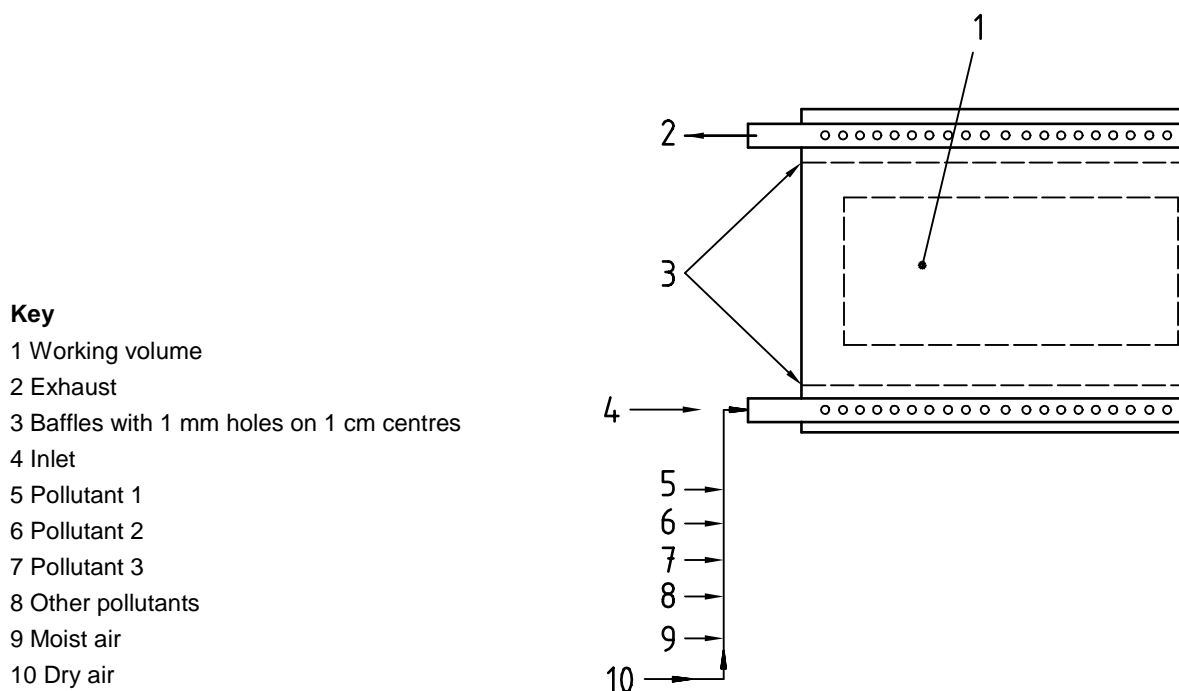
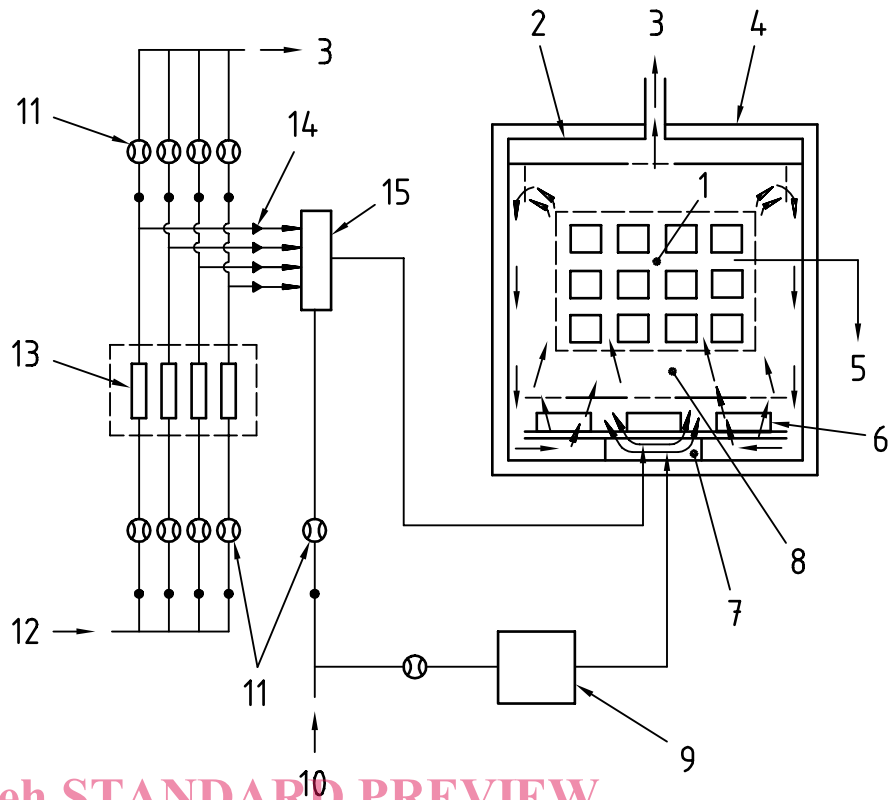


Figure 1 — Schematic flow through mixed flowing gas (MFG) test system

Key

- 1 Working volume
- 2 Test chamber insert
- 3 Exhaust
- 4 Temperature controlled chamber
- 5 Gas monitoring meters
- 6 Fans
- 7 Mixed gas / humidified air blending area
- 8 Main flow
- 9 Humidity source
- 10 Air
- 11 Flow meters
- 12 Nitrogen
- 13 Permeation tubes
- 14 Limiting orifices
- 15 Mixing chamber



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Figure 2 — Schematic vertical recirculating mixed flowing gas (MFG) test system

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Key

- 1 Gas concentration measurement points
- 2 Temperature and humidity measurement
- 3 Gas velocity measurement
- 4 Test sample region
- 5 Baffles
- 6 Gas concentration analysis
- 7 Test sample data collection
- 8 Chamber exhaust
- 9 Mixed gas supply
- 10 Fans
- 11 Air flow direction
- 12 Humidity source
- 13 Multiple gas sources
- 14 Make-up air
- 15 Gas mixing manifold
- 16 Temperature control chamber
- 17 Corrosive gas chamber

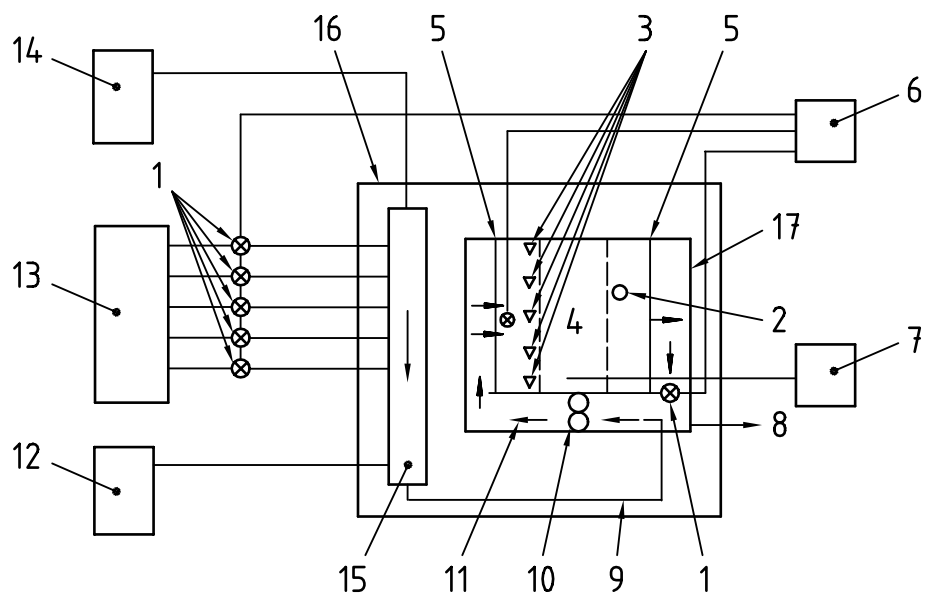


Figure 3 — Schematic horizontal recirculating mixed flowing gas (MFG) test system

5.3 Gas supply system

5.3.1 Description and requirements

The gas supply system consists of five main parts:

- a source of clean, dry, filtered air;
- a humidity source;
- corrosive gas source(s);
- gas delivery system;
- corrosive gas concentration monitoring system(s).

Total supply capacity shall be such as to meet requirements for control of gas concentrations. The minimum number of volume changes is determined by the requirement that the concentration of corrosive gases be maintained within $\pm 15\%$ between gas inlet and outlet. This is verified by measurement of the gas concentrations near the gas inlet upstream of the usable chamber working volume and comparing with gas concentrations measured downstream of the usable chamber working volume just prior to the chamber exhaust. These values shall be within $\pm 15\%$ (see 6.3). Alternative methods of demonstrating compliance with the maximum allowable concentration gradient are acceptable. Normally a conditioned chamber equilibrates within several hours of sample loading and start of corrosive gas supply. Times longer than 2 h shall be reported in the test report; see clause 7. A guide to estimating supply requirements is provided in annex B.

5.3.2 Clean, dry, filtered air source (standards.iteh.ai)

Gases other than oxygen and nitrogen that are present in the dry air source shall be less than or equal to those defined by OSHA Class D limits. In addition, gases other than nitrogen, oxygen, carbon dioxide, noble gases, methane, nitrous oxide and hydrogen shall be less than $0,005 \text{ (cm}^3/\text{m}^3\text{)}$ by volume total and shall be high efficiency particulate arrestant (HEPA) filtered.

5.3.3 Humidity source

The humidity source shall use distilled or deionized water, of ISO 3696 grade 1 or better, and shall introduce no extraneous material. The humidity source shall be maintained equivalent to ISO 3696 grade 2 or better, with the exception that electrical resistivity shall be maintained equivalent to ISO 3696 grade 3. The time-averaged value of humidity shall be within $\pm 1\%$ relative humidity of the specified value with absolute variations no greater than $\pm 3\%$ relative humidity from the specified value.

Table 1 — Instrumental methods for gaseous components

Gas	Suitable instrumental method	Suitable procedure
H ₂ S	Photometric or luminescence	Commercial equipment is available
SO ₂	Photometric or luminescence	ISO 4221, ISO 6767, ISO 6349, ISO 7934. Commercial equipment is available
NO ₂	Chemiluminescence	ISO 7996. Commercial equipment is available
Cl ₂	Electrochemical	ISO 10313
NOTE	The instrument manufacturer's instructions for delivering samples to the instrument should be followed.	

5.3.4 Corrosive gas sources

Corrosive (test) gases, such as nitrogen dioxide, hydrogen sulfide, chlorine, sulfur dioxide etc. shall be of chemically pure grade or better. Such gases are frequently supplied in a carrier gas such as nitrogen which shall be of pre-purified grade or better.

5.3.5 Gas delivery system

The gas delivery system is comprised of three main parts: gas supply lines, gas control valves and flow controllers, and a mixing chamber. The test delivery system shall be capable of delivering gases at the required concentrations and rates within the test chamber.

- a) All materials used for the gas transport system shall not interact with the gases to the extent that chamber gas concentrations are affected.
- b) Gases, make-up air and water vapour shall be thoroughly mixed before gas delivery to the samples under test in the chamber. Care shall be taken to ensure absence of aerosol formation in the mixing chamber whereby gases are consumed in the formation of particulates that can interfere with gas concentration control and can introduce corrosion processes that are not representative of gaseous corrosion mechanisms. Aerosol formation can be detected by the presence of a visible film or deposit on the interior surface of the gas system where the gases are mixed.
- c) Any fogging of the tubing walls or mixing chamber walls can be taken to be an indication of a loss of corrosive gases from the atmosphere. Final mixing of the specified gases shall occur inside a separate area of, or as close as possible to, the test chamber so as to ensure thermal equilibration with the test chamber.
- d) There shall be a means for measuring the flow at the inlet of the chamber and also at the exhaust of negative pressure chambers to ensure the absence of uncalibrated gas streams.

5.3.6 Corrosive gas concentration monitoring system

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Standard measurement systems for very low level gas concentrations are listed in Table 1, which provides for gases in common use in present mixed flowing gas systems, for testing electrical contact performance.

- a) Each instrument shall be characterized for interference with the gases specified, both individually and mixed.
- b) Depending on the exact equipment set used, it may not be possible to measure accurately the concentration of some gases, such as chlorine, in combination with any of the other gases.
- c) The analytical instruments shall be maintained and calibrated electronically in accordance with the manufacturers' recommendations. Standard gas sources shall be calibrated in accordance with the manufacturers' specifications. Analysers shall be calibrated to standard gas sources in accordance with the manufacturers' recommendations. They shall be calibrated before and after each test and whenever the indicated concentration changes exceed the permitted variation in the test specification.

NOTE If the chlorine monitor is not being used during the test, it need not be calibrated during the test.

- d) Control of the humidity within the test chamber itself is part of the chamber monitoring system which is described in 5.4.

5.4 Chamber monitoring system

5.4.1 General

Chamber monitoring systems shall be used to ensure test reproducibility from one test run to the next. Monitoring instruments shall be calibrated periodically because the corrosive effects of mixed gas environments can affect instrument sensitivity and accuracy. The chamber monitoring system shall address four test parameters: temperature, humidity, gas concentrations and corrosivity.