Standard Practice for
Conducting Mixed Flowing Gas (MFG) Environmental Tests[1]

1. Scope

1.1 This practice provides procedures for conducting environmental tests involving exposures to controlled quantities of corrosive gas mixtures.

1.2 This practice provides for the required equipment and methods for gas, temperature, and humidity control which enable tests to be conducted in a reproducible manner. Reproducibility is measured through the use of control coupons whose corrosion films 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.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to become familiar with all hazards including those identified in the appropriate Material Safety Data Sheet for this product/material as provided by the manufacturer, to establish appropriate safety and health practices, and determine the applicability of regulatory limitations prior to use.* See 5.1.2.4.

2. Referenced Documents

2.1 *ASTM Standards:*

B 542 Terminology Relating to Electrical Contacts and Their Use[2]

B 765 Guide for Selection of Porosity Tests for Electrodeposits and Related Metallic Coatings[3]

B 808 Test Method for Monitoring of Atmospheric Corrosion Chambers by Quartz Crystal Microbalances[2]

B 810 Test Method for Calibration of Atmospheric Corrosion Test Chambers by Change in Mass of Copper Coupons[2]

B 825 Test Method for Coulometric Reduction of Surface Films on Metallic Test Samples[2]

B 826 Test Method for Monitoring Atmospheric Corrosion Tests by Electrical Resistance Probes[2]

B 845 Guide for Mixed Flowing Gas (MFG) Tests for Electrical Contacts[2]

D 1193 Specification for Reagent Water[4]

D 1607 Test Method for Nitrogen Dioxide Content of the Atmosphere (Griess-Saltzman Reaction)[5]

D 2912 Test Method for Oxidant Content of the Atmosphere (Neutral KI)[6]

D 2914 Test Methods for Sulfur Dioxide Content of the Atmosphere (West-Gaeke Method)[5]

D 3449 Test Method for Sulfur Dioxide in Workplace Atmospheres (Barium Perchlorate Method)[6]

D 3464 Test Method for Average Velocity in a Duct Using a Thermal Anemometer[5]

D 3609 Practice for Calibration Techniques Using Permeation Tubes[5]

D 3824 Test Methods for Continuous Measurement of Oxides of Nitrogen in the Ambient or Workplace Atmosphere by the Chemiluminescent Method[5]

D 4230 Test Method of Measuring Humidity With Cooled-Surface Condensation (Dew-Point) Hygrometer[5]

E 902 Practice for Checking the Operating Characteristics of X-Ray Photoelectron Spectrometers[7]

G 91 Practice for Monitoring Atmospheric SO_2 Using Sulfation Plate Technique[8]

3. Terminology

3.1 Definitions relating to electrical contacts are in accordance with Terminology B 542.

4. Significance and Use

4.1 Mixed flowing gas (MFG) tests are used to simulate or amplify exposure to environmental conditions which electrical

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contacts or connectors can be expected to experience in various application environments (**1, 2**).[9]

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

4.3 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 platings (**1, 2**).

4.4 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 in the MFG test. A guide to the selection of simulation conditions suitable for a variety of environments is found in Guide B 845.

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

4.6 The MFG tests are useful for connector systems whose contact surfaces are plated or clad with gold or other precious metal finishes. For such surfaces, environmentally produced failures are often due to high resistance 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 and corrosion product migration or tarnish creepage from pores in the precious metal coating and from unplated base metal boundaries, if present.

4.7 The MFG exposures can be used to evaluate novel electrical contact metallization for susceptibility to degradation due to environmental exposure to the test corrosive gases.

4.8 The MFG exposures can be used to evaluate the shielding capability of connector housings which may act as a barrier to the ingress of corrosive gases.

4.9 The MFG exposures can be used to evaluate the susceptibility of other connector materials such as plastic housings to degradation from the test corrosive gases.

4.10 The MFG tests are not normally used as porosity tests. For a guide to porosity testing, see Guide B 765.

4.11 The MFG tests are generally not applicable where the failure mechanism is other than pollutant gas corrosion such as in tin-coated separable contacts.

5. Apparatus

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

5.1.1 *Corrosion Test Chamber*:

5.1.1.1 The chamber shall consist of an enclosure made of nonreactive, low-absorbing, nonmetallic materials contained within a cabinet or oven capable of maintaining the tempera-

ture to a maximum tolerance of $\pm 1°C$ with a preferred tolerance held to $\pm 0.5°C$ within the usable chamber working space accordance with 7.3, with a means to introduce and exhaust gases from the chamber.

5.1.1.2 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.

5.1.1.3 The chamber shall have provision for maintaining uniformity of the average gas flow velocity within ± 20 % of a specified value or of the chamber average when the chamber is empty. For chambers with a dimension of more than 0.5 m, measurement points shall be in accordance with Test Method B 810. 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 measurements shall be repeated a second time. After the two sets of measurements are recorded, a third complete set shall be recorded. The arithmetic average of the 15 or more measurements shall be the chamber average. See 7.5 and 7.6.8. If a hot wire anemometer is used for gas velocity measurements, it shall be made in accordance with Test Method D 3464, with the exception that sample sites shall be in accordance with Test Method B 810.

5.1.1.4 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 (**3, 4**). Provision for controlling the test illumination level in accordance with a test specification shall be made.
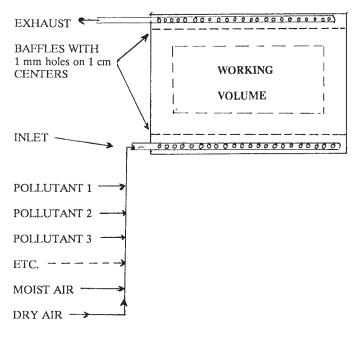
5.1.1.5 Examples of test chamber systems are diagrammed in Figs. 1-3. They are not to be considered exclusive examples.

5.1.2 *Gas Supply System*:

5.1.2.1 *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; and corrosive gas concentration monitoring system(s). Total supply capacity must 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 ± 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 ± 15 % (see 7.6). Alternative methods of demonstrating compliance with the maximum allowable concentration gradient are acceptable. Normally, a conditioned chamber equilibrates within several hours after sample loading and start of the corrosive gas supply. Times longer than 2 h shall be reported in the test report; see Section 8. A guide to estimating supply requirements is provided in Appendix X1.

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[9] The boldface numbers in parentheses refer to the list of references at the end of this standard.

LAMINAR FLOW - SINGLE PASS

MFG Corrosion Chamber with

Distributed Inlet and Exhaust

FIG. 1 Schematic Flow-Through Mixed Flowing Gas (MFG) Test System

NOTE 1—Guidance: when inlet to outlet concentrations vary by more than ±15 %, it usually indicates an overloaded chamber.

5.1.2.2 *Clean, Dry, Filtered Air Source*— Gases other than oxygen and nitrogen that are present in the dry air source shall be less than or equal to those defined by OHSA Class D limits with the following additional constraint. Gases other than nitrogen, oxygen, carbon dioxide, noble gases, methane, nitrous oxide, and hydrogen shall be less than 0.005 (ppm) by volume total and shall be High Efficiency Particulate Arrestants (HEPA) filtered.

5.1.2.3 *Humidity Source*—The humidity source shall use distilled or deionized water, Specification D 1193, Type 1 or better, and shall introduce no extraneous material. The humidity source shall be maintained equivalent to Specification D 1193 Type II or better, with the exception that electrical resistivity shall be maintained equivalent to Specification D 1193 Type IV. The time averaged value of humidity shall be within ±1 % relative humidity of the specified value with absolute variations no greater than ±3 % relative humidity from the specified value.

5.1.2.4 *Corrosive Gas Sources*—Corrosive (test) gases, such as nitrogen dioxide, hydrogen sulfide, chlorine, sulfur dioxide, etc. shall be of chemically pure[10] grade or better. Such gases are frequently supplied in a carrier gas such as nitrogen which shall be of Pre-Purified [10] grade or better. (**Warning**—

This practice involves the use of hazardous materials, procedures, and equipment. The gas concentrations in the test chamber may be within permissible exposure limits (PEL).[11] However, concentrations in the compressed gas cylinders or permeation devices are often above the PEL, and may exceed the immediately dangerous to life and health level (IDHL). This practice does not address safety issues associated with MFG testing.)

5.1.2.5 *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 gas delivery system shall be capable of delivering gases at the required concentrations and rates within the test chamber.

(1) All materials used for the gas transport system must not interact with the gases to the extent that chamber gas concentrations are affected.

(2) Gases, make-up air, and water vapor must be thoroughly mixed before gas delivery to the samples under test in the chambers. Care must be taken to ensure absence of aerosol formation in the mixing chamber whereby gases are consumed in the formation of particulates which may interfere with gas concentration control and may introduce corrosion processes which are not representative of gaseous corrosion mechanisms. Aerosol formation may be detected by the presence of a visible film or deposit on the interior surface of the gas system where the gases are mixed.

(3) 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 should 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.

(4) Flow measurement capability is required at the inlet of the chamber and also at the exhaust of negative pressure chambers to ensure the absence of uncalibrated gas streams.

5.1.2.6 *Corrosive Gas Concentration Monitoring System*— 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.

(1) Each instrument must be characterized for interference with the gases specified, both individually and mixed.

(2) Depending on the exact equipment set used, it may not be possible to accurately measure the concentration of some gases, such as chlorine, in combination with any of the other gases.

(3) The analytic instruments shall be maintained and calibrated electronically in accordance with the manufacturers' recommendations. Standard gas sources shall also be calibrated in accordance with the manufacturers' specifications, or in accordance with Practice D 3609. Gas concentration analyzers shall be calibrated to standard gas sources in accordance with the manufacturers' recommendations. They shall be calibrated

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[10] Chemically Pure and Pre-Purified are designations of Matheson Gas Co., East Rutherford, NJ, for specific grades of purity of gas. Other vendors such as AIRCO have equivalent gas purities available sold under different terminology.

[11] Pocket Guide to Chemical Hazards, National Institute for Occupational Safety and Health, U.S. Department of Health and Human Service, Publication #85-114, fifth printing.
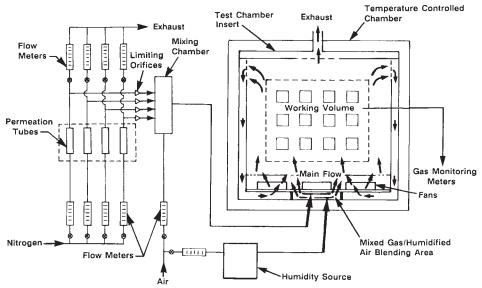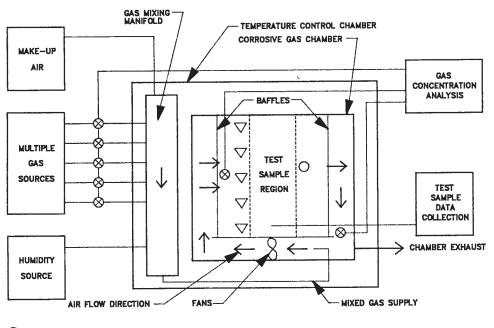
FIG. 2 Schematic Vertical Recirculating Mixed Flowing Gas (MFG) Test System

⊗ GAS CONCENTRATION MEASUREMENT POINTS

○ TEMPERATURE AND HUMIDITY MEASUREMENT

▽ GAS VELOCITY MEASUREMENT

FIG. 3 Schematic Horizontal Recirculating Mixed Flowing Gas (MFG) Test System

before and after each test and whenever the indicated concentration changes exceed the allowed variation in the test specification.

(4) Control of the temperature and humidity within the test chamber itself is part of the chamber monitoring system which is described in 5.1.3

NOTE 2—If the chlorine monitor is not being used during the test, it need not be calibrated during the test.

5.1.3 *Chamber Monitoring System*—Chamber monitoring systems are required to ensure test reproducibility from one test run to the next. Calibration of monitoring instruments is required periodically because the corrosive effects of mixed gas environments can affect instrument sensitivity and accuracy. The chamber monitoring system must address four test parameters: temperature, humidity, gas concentrations, and corrosivity.

5.1.3.1 *Temperature Monitoring*—Temperature monitoring is usually a simple thermocouple or other temperature measurement device capable of the required resolution of 0.2°C and accuracy of ±0.5°C within the temperature range required by the test specification. For test temperatures above 40°C, see 7.6.5.

TABLE 1 Instrumental Methods for Gaseous Components

NOTE 1—Commercial equipment such as Monitor Labs 8770, Hydrogen Sulfide Converter, in conjunction with Monitor Labs 8850, Sulfur Dioxide Analyzer is suitable for this purpose.

NOTE 2— Commercial equipment such as Monitor Labs 8850, Sulfur Dioxide Analyzer is suitable for this purpose.

NOTE 3—Commercial equipment such as Monitor Labs 8840, Nitrogen Oxides Analyzer is suitable for this purpose.

Gas	Suitable Instrumental Method	Suitable Procedure
H_2S	Photometric or luminescence	See Note 1
SO_2	Photometric or luminescence	Test Methods D 2914, G 91, D 3449, see Note 2
NO_2	Chemiluminescence	Method D 3824, see Note 3
Cl_2	Electrochemical or Reflectometric	Test Method D 2912

The instrument manufacturer's instructions for delivering samples to the instruments should be followed.

5.1.3.2 *Humidity Monitoring*—Humidity must be determined by an apparatus with a resolution of 0.5 % relative humidity and an accuracy of ±1 % relative humidity. Test Method D 4230 describes a dew point method which meets this requirement. For test temperatures above 40°C, see 7.6.5.

5.1.3.3 *Corrosive Gas Monitoring*—Chamber corrosive gas concentration monitoring must be accomplished by provision of sampling lines from the test chamber to the gas concentration analyzers. These sampling lines must be maintained above the chamber dew point temperature. The interior of the gas concentration analyzers shall also be maintained above the chamber dew point temperature. For test temperatures above 40°C, see 7.6.5.

5.1.3.4 *Chamber Corrosivity Monitoring*— Chamber corrosivity monitoring can be accomplished by a number of complementary techniques, none of which provide both a comprehensive analysis of the corrosion process and an instantaneous indication of the corrosion rate. Five acceptable techniques are as follows: metal coupon corrosion mass gain, corrosion film analysis by coulometric reduction, corrosion film analysis by electron or X-ray beam analysis, quartz crystal microbalance mass gain, and electrical resistance measurement of corroding metal conductors (see Note 3). The first three provide information subsequent to the test whereas the last two can be used in situ in the test chamber to provide information during the test itself. See Appendix X2 for a discussion of these methods. It is recommended that the test requester specify chamber corrosivity monitoring methods to be used.

NOTE 3—A potential sixth method utilizing porous gold coupons is under investigation.

5.1.4 *Chamber Operating System*—The chamber operating system is comprised of equipment and software necessary to adequately control all of the variables of the test. This will include data logging and alert procedures for operation outside of desired bounds. Some form of computer control is highly recommended to assure satisfactory operation during unattended periods and for data tracking for failure analysis in case the test is disrupted.

6. Reagents and Materials

6.1 Materials required to conduct flowing mixed gas tests are as follows:

6.1.1 *Purity of Water*—Water for humidity generation shall be equivalent to Type 1 or better of Specification D 1193.

6.1.2 *Carrier Gas*—Carrier gas such as nitrogen shall not introduce reactive constituents into the test atmosphere to an amount of more than 5 % of any specified corrosive test atmosphere constituent.

6.1.3 *Clean Filtered Air*—Clean filtered air as required for makeup to support the necessary exchange rate, in accordance with 7.6.7.1 (*2*) is specified in 5.1.2.2.

6.1.4 *Corrosive Gases*—Corrosive gases shall be chemically pure[9] grade or equivalent.

6.1.5 *Corrosivity Monitor Materials (CMM)*—CMM are comprised of the coupons that are exposed to the test atmosphere for mass gain or coulometric reduction in accordance with Test Methods B 810 and B 825, respectively, the coated quartz crystals used for microbalance measurements in accordance with Test Method B 808, resistance monitor materials in accordance with Test Method B 826, or other coupons for analytical techniques described in Appendix X2.3.

7. Procedure

7.1 The following procedure is comprised of requirements and other comments provided as a general guide to achieving reproducible results with MFG testing. This procedure is compatible with most test facilities; however, differences in apparatus, test conditions, or local safety requirements may necessitate alternative procedures. Any deviations shall be reported with all test results (see Section 8).

7.2 The procedure is comprised of the following major activities: test chamber calibration, sample preparation, test chamber set-up, test chamber start-up, test chamber operation during test duration, test chamber shut-down, and reporting requirements.

7.3 *Test Chamber Calibration*—The spatial uniformity of the corrosivity of test chambers larger than 0.5 m on a side shall be measured in accordance with Test Method B 810, which describes the required placement scheme for calibration samples which are used to determine corrosion rate uniformity over the entire chamber volume. For chambers smaller than 0.5 m on a side or chambers of unusual geometry, use sufficient samples for corrosivity characterization so as to clearly delineate the usable chamber working volume as defined in this paragraph. This profiling shall be done when the chamber is initially built and after any structural change to the chamber that may affect the flow of test gas over the test samples.Test Method B 810 describes a procedure using mass gain. Alternative means to characterize corrosion rates such as Test Method B 825, Coulometric Reduction, or Test Method B 808, Quartz Crystal Microbalance, in accordance with 5.1.3.4 are also acceptable. A minimum of three corrosivity monitors of a given type must be used, if possible, in each chamber location. The average corrosivity for that location must be within 15 % of the average for the entire chamber. When a single monitor has to be used at a location, due to chamber size limitations or monitor geometry, the average corrosivity for that location must be based upon three consecutive calibration runs. These requirements define the usable chamber working space.

NOTE 4—Profiling does not remove the necessity to provide and

evaluate CMM for each test run of the test chamber.

7.4 *Sample Preparation*—Two types of samples are used for these tests, CMM and the test samples being evaluated. Prepare CMM in accordance with their respective standards, such as Test Method B 810.

7.4.1 Prepare the test samples in accordance with any agreement between vendor and user of the samples being tested. Such preparation shall be consistent with normal preparations expected when test samples would be exposed to normal application environments in their intended applications except when evaluation of preparation methods is the object of the test.

7.5 *Test Chamber Set-Up*—Place test samples and CMM in the chamber in a manner that is representative of the way the test samples would be used in the application environment, if known. This should be done in a consistent manner, such that the test results will be reproducible over time.

7.5.1 In general, the samples shall be suspended or held with their long dimension parallel to the flow of air and a minimum of 5 cm away from any surface to avoid boundary layer effects. It is particularly important that no test samples or CMM be shielded from the source of the pollutant gases by any control coupon, test fixture, test samples, test rack, or other obstruction placed upstream.

7.5.2 In general, when larger systems under test are being expressed to a MFG environment, the interior of the system under test will be underexposed due to the gettering or reaction of pollutant gases by the surrounding system surfaces. Under these circumstances, the system should be placed in the exposure chamber in a configuration that is consistent with exposure in actual field configuration. CMMs should be placed around the system under test.

7.6 *Test Chamber Start-Up*:

7.6.1 *Test Conditions*—Test conditions such as those given in Section 8 shall be specified by the test requester. Test Method B 845 is a guide to selection of such conditions for specification purposes.

7.6.2 *Avoidance of Condensation*—Establish an apparatus specific start-up and shut-down procedure to avoid visible water condensation on the test samples and CMM at time of insertion into the chamber. Such condensation on the parts invalidates the test. To avoid condensation at start-up the parts under test shall be at a temperature that is greater than the dew point temperature of the chamber at insertion time. To avoid condensation at shut-down the laboratory temperature shall be greater than the dew point temperature of the exposure chamber at sample removal time.

7.6.3 *Chamber Preparation*—When contaminants such as condensed gases (for example, free sulfur or organic material from test samples) or corrosion particulate deposits are present or suspected, clean the inside of the chamber to reduce the concentration of adsorbed gases by wiping the interior walls with a clean, lint-free cloth before installing samples at the start of any test. Residual contamination can affect the accuracy of subsequent chlorine measurements.

7.6.3.1 An indication of the need for a wipe down would be an abnormally long time (in excess of 20 chamber gas exchanges for low-sulfur (for example, <0.020 ppm H_2S) tests

or in excess of 200 chamber gas exchanges for high-sulfur tests) to reach 10 % or 0.001 ppm corrosive gas concentration levels after chamber shutdown.

7.6.4 *Chamber Loading*:

7.6.4.1 Place the test samples and control materials into the chamber when the samples, materials, and chamber are at ambient temperature and relative humidity in order to avoid visible condensation. Alternatively, samples at chamber temperature can be placed directly into a heated chamber at or below specified humidity.

7.6.4.2 For tests which require in situ measurements on the test samples, install necessary electrical access cabling at this time and make initial measurements, as required by the test specification.

7.6.5 *Chamber Heating*—The practical upper limit to the test temperature for this procedure is determined by the internal temperature of the analyzers, including any auxiliary heating, such that condensation of the sampled gas stream will not occur within the instrument. In order to avoid condensation in the analytical instruments and sampling lines, the relative humidity of the sampled gas mixture in the sampling lines and analytical instruments shall not exceed 80 % relative humidity. This is generally accomplished by heating the sampling lines and instruments as required. Manufacturers of the analyzers should be consulted to determine maximum temperatures at which the analyzers can be maintained. Modifications of this procedure such as limiting chamber humidity to a wet-bulb temperature less than the instrument internal temperature during corrosive gas supply settings and subsequent elevation of humidity to specified values may be required for high temperature (for example, 70°C), high-humidity corrosive gas tests.

7.6.5.1 Heat the chamber to the specified test temperature, if required. A holding time of at least 1 h is recommended to ensure temperature equilibration of the test samples. A longer time may be necessary for massive assemblies.

7.6.6 *Chamber Humidification*:

7.6.6.1 Increase the chamber humidity to the specified test relative humidity, if required.

7.6.7 *Gas Level Setting*:

7.6.7.1 Confirm that temperature and humidity are at equilibrium at specified test conditions.

7.6.7.2 When conducting multiple gas tests with chlorine as one of the corrosive gases, chlorine must be the first gas whose supply setting is established. This is because of interferences from other gases which are due to present limitations of the chlorine gas monitoring equipment in common use. Allow the chlorine levels to come to equilibrium in the chamber for at least 1–2 h.

7.6.7.3 Introduce all the other corrosive gases to the level specified and measure the gas concentrations (see 5.1.2.6), in the test chamber in accordance with 5.1.3.3; adjust the gas supply rates and volume exchange rate until the downstream gas concentrations are within ±15 % or 0.003 ppm, whichever is larger, of the upstream concentrations and at the gas concentrations specified. Allow to stabilize and repeat measurement after 1 to 2 h to confirm gas supply and volume

exchange rate setting. Circulating fans shall be running during this gas supply setting in recirculating-type chambers.

7.6.7.4 The following points concerning gas concentrations should be noted.

(1) Inability to achieve 15 % (0.003 ppm) gas concentration tolerance between upstream and downstream values may indicate insufficient exchange rate or excessive loading of test samples.

(2) Once gas levels are set initially, any change in the gas supply system requires confirmation of compliance with the requirements of 7.6.7.1 *(2)* and *(3)* and may necessitate resetting of the gas controls; any such actions shall be reported in Section 8.

(3) For assembly level testing, for example, a disk drive assembly or large wiring harness, gas levels should be set upstream of the test object at the maximum exchange capacity of the system, with the assembly in place. Record the downstream gas concentrations and report them in the test report as a deviation, see Section 8. When testing at assembly level, the requirement that the gas concentration in the exhaust stream be within 15 % (or 0.003 ppm, whichever is larger) of the inlet stream may not be applicable, since it is entirely possible that under operating conditions, exhaust streams from the assembly may be depleted of pollutants due to absorption within the assembly itself. It is recommended that the test requester and test operator discuss the expected deviation from the 15 % concentration variation for assembly testing.

7.6.8 *Air Velocity Confirmation*—For tests specifying air velocity, after test samples are placed in the chamber, and gas levels and exchange rates are set, measure the velocity of the corrosive gas air stream impinging on the test samples between 5 and 8 cm upstream of the test samples for compliance with the air velocity specified and the allowable tolerance of ±20 %.

7.6.9 *Corrosivity Setting*—Where corrosivity is required to be monitored by means of one or more in situ continuous monitors such as a quartz crystal microbalance (QCM) or resistance monitor (RM), maintain the corrosivity within the bounds specified by the test specification. Deviations from the expected corrosivity require immediate attention to the controlling test parameters such as temperature, humidity, and gas concentration to rectify the deviation. Report the inability to attain the specified corrosivity at the specified test sample loading with all other parameters in the specified range as a test deviation in Section 8.

7.6.10 *Test Duration*—Test duration can be specified by two different means. The test may be specified to endure a set period of time, or it may be specified to endure until a required total corrosion, as measured by an in situ corrosivity monitor, is achieved.

7.7 *Test Chamber Operation*—Monitor the test chamber for temperature, humidity, and pollutant gas concentrations to demonstrate chamber stability with respect to short-term fluctuations and long-term drifts. Place CMM in the test chamber adjacent to the test samples. This will provide a measure of chamber corrosivity after the test is completed. For a plane array of test samples place a minimum of five CMM, one at each corner and one at the center of the array of test samples.

Corrosivity monitors such as resistance monitors or quartz crystal microbalances are recommended to provide an integrated continuous assessment of chamber status.

7.7.1 *Test Tolerances*—Maintain the following tolerances on test parameters unless otherwise specified by the test requester:

7.7.1.1 Maintain the temperature within ±1°C with a preferred tolerance of ±0.5°C.

7.7.1.2 Maintain the humidity at an average value within ±1 % relative humidity with an absolute variation less than 3 % relative humidity.

7.7.1.3 Maintain the gas concentrations within ±15 % or ±0.003 ppm, whichever is larger.

7.7.1.4 If specified, maintain the corrosivity within 15 % of the specified value.

7.7.1.5 Maintain the test duration within ±2 % or ±2 h, whichever is longer.

7.7.2 *Psychrometric Monitoring*—Continuous or periodic monitoring of temperature and humidity is required. The maximum period between measurements shall be 30 min.

7.7.2.1 Air velocity need not be monitored during the test unless significant changes in sample placement occur during the test. Some means of verifying that the fans are operating properly is required in recirculating-type chambers in order to ensure that air velocity remains within the tolerance band specified or the range for which chamber calibration was obtained.

7.7.3 *On-Line Control*—In addition to monitoring, some type of on-line control is recommended. This allows adjustments to be made in the gas concentrations dynamically, to increase the probability of a valid test.

7.7.4 *Test Continuity*—The test exposure should be run continuously with as few interruptions as possible, unless otherwise specified. Interruptions for removal or replacement of test samples or CMM, during which time chamber conditions may vary outside of limits defined in 7.7.1, shall not be considered deviations as long as total duration of all interruptions is less than 5 % of total test time.

NOTE 5—These deviations can be minimized by building a small door within the main chamber access door to facilitate the removal or addition of CMM.

7.7.4.1 *Test Integrity*—The test shall not be disrupted by the addition of new samples for a different test during the operation of the test. New samples introduce fresh absorbing surfaces which can significantly alter the gas concentrations at which the original samples were being tested; such a disruption would lead to problems reproducing test results and is unacceptable.

7.8 *Test Chamber Shut-Down*:

7.8.1 *Electrical Power-Down*—Discontinue electrical power to any devices under test and to in situ corrosivity monitors.

7.8.2 *Corrosive Gas Shut-Down*—Discontinue corrosive gas supply, except for chlorine, if used. Allow chlorine level to equilibrate in the absence of the other gases. Measure chlorine level (also, measure residual levels of other gases) to ensure compliance with 7.6.7.1 *(2)* and report if it is outside the test specification. Then, discontinue chlorine supply.

7.8.2.1 If high levels of H_2S of SO_2, or both, are used, it may not be possible to accurately measure the Cl_2 concentration because the sulfur species emitted from the test samples can interfere in a negative manner to reduce the oxidant-caused signal in some chlorine monitors (for example, MAST Oxidant Monitor). If such interference is suspected, it is necessary to remove the test samples prior to verifying the chlorine concentration. Empty the test chamber of test samples and CMM. Reseal the test chamber and restart the chlorine flow at the prior setting. After equilibration of chlorine, measure the chlorine level and record for inclusion in Section 8. Discontinue chlorine flow.

NOTE 6—If the chlorine level is close to or above the accepted time weighted average (TWA), the testor will have to record the chlorine flow settings and shut off all corrosive gas supply before opening the chamber to the laboratory. The current accepted TWA for chlorine is 500 parts per billion. It is good laboratory practice to minimize any personnel exposure to corrosive gases.[12]

7.8.3 *Humidity Control*—Discontinue humidity generation while maintaining chamber temperature, if it is necessary to reduce chamber temperature. When the relative humidity has stabilized at a low level, the chamber temperature may be reduced in convenient increments while ensuring freedom from condensation until the chamber can be safely opened and test samples and CMM removed from the chamber.

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[12] 1995–1996 Threshold Limit Values (TLVs) for Chemical Substances and Physical Agents and Biological Exposure Indices (BEIs), American Conference of Governmental Industrial Hygienists, Technical Affairs Office, 1330 Kemper Meadow Drive, Cincinnati, OH, 45240.

8. Report

8.1 The report shall contain the following information:

8.1.1 Facility name.

8.1.2 Test engineer.

8.1.3 Test requester.

8.1.4 Date.

8.1.5 *Test Samples*—Description; number of test samples; condition tested; exposure intervals; and data summary.

8.1.6 *Corrosivity Monitor Materials (CMM)*—Description (each type); number of CMM; description of CMM placement; exposure intervals; data from CMM; procedures used for preparation and analysis (for example, in accordance with Test Method B 810); and equilibration time to stabilize gases at ±15 % (or 0.003 ppm), inlet to exhaust, if longer than 2 h.

8.1.7 *Test Conditions: Levels and Relevant Tolerances*—Gas concentrations; temperature and humidity; air velocity, direction; illumination condition; exchange rate; and test duration.

8.1.8 Chamber dimensions.

8.1.9 Usable chamber working space in accordance with 7.3.

8.1.10 Deviations from normal conditions.

8.1.11 Record of all interruptions (reason and duration).

9. Keywords

9.1 air velocity; chlorine; corrosion; corrosive gas testing; corrosivity; corrosivity monitor; coulometry; environmental; humidity; hydrogen sulfide; mixed flowing gas; nitrogen oxide; pollutant; pore corrosion; quartz crystal microbalance; resistance monitor; sulfur; sulfur dioxide; tarnish; temperature; testing

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APPENDIXES

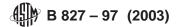### (Nonmandatory Information)

X1. ESTIMATING REQUIRED CORROSIVE GAS EXCHANGE RATE

X1.1 The required rate of corrosive gas exchange can be estimated from the total gas consumption required to obtain the expected corrosion rate for the test being performed. In a typical MFG test the initial corrosion rate can be as high as 8 nm of copper corrosion film growth/h over the first 8 h of the test. The most conservative assumption of one atom of corrosive gas per atom of copper implies that such a film thickness requires 2×10^{16} chlorine atoms/h/cm² of exposed copper surface, if the entire film is comprised of CuCl. Thus the amount of chlorine supplied to the test chamber must be 2×10^{17}/h/cm² if no more than 10 % loss of concentration can be accepted downstream of the exposed copper surface in accordance with the requirements of this practice. If the chlorine is being supplied at a concentration of 0.01 ppm in carrier gas, then 0.83 m³ of such supply is required/h/cm² of exposed copper surface. For 100 cm² of exposed copper, the gas supply must be 83 m³/h.

X1.2 If half of the film is oxide and half is basic copper chloride, $Cu_2(OH)_3Cl$, as is more likely, then the chlorine consumption rate is reduced by a factor of 4.7 to 17.7 m³/h/100 cm² of exposed copper.

8

X2. CORROSIVITY MONITORING METHODS

X2.1 *Mass Gain Coupons*—Test Method B 810 describes a technique for use of copper coupons in chamber monitoring which utilizes mass gain due to formation of corrosion products from interaction of the corrosive gases with the exposed surface of the copper coupon. It describes coupon cleaning, handling, placement, and evaluation procedures.

X2.2 *Coulometric Reduction*—Test Method B 825 describes a procedure for determining the relative amounts of different corrosion film constituents in corrosion films formed on copper and silver coupons by means of coulometric reduction techniques. The technique also provides a measure of the total amount of copper or silver which has reacted to form corrosion products on the surface of the coupons which have been exposed in the test chamber.

X2.3 *Surface Analysis*—Surface analysis of corrosion films has been performed by a number of analytical techniques including X-ray diffraction, X-ray emission spectroscopy, X-ray photoelectron spectroscopy (see Practice E 902), Auger electron analysis and secondary ion mass spectroscopy. All of these techniques yield different data which can be correlated to develop a more complete understanding of corrosion behavior. These techniques are more important when metals other than copper or silver are being examined for susceptibility to mixed flowing gas testing because of the absence of extensive data bases on those other metals in the environments considered here.

X2.4 *Quartz Crystal Microbalance*—Test Method B 808 describes the use of the quartz crystal microbalance to provide a real time monitor of the corrosion rate of a chamber. The technique is based on a frequency measurement of a resonating quartz crystal which has been coated with a thin film of reactive metal such as copper. As the copper corrodes, the mass of the crystal plus copper plus corrosion product increases leading to a smaller resonant frequency. The frequency shift is directly related to the amount of corrosion and is sensitive to less than a monolayer of corrosion product. This sensitivity provides an immediate measure of corrosion rate which can be related to gas concentrations or other chamber conditions such as temperature or humidity.

X2.5 *Resistance Monitoring*—Test Method B 826 describes the use of a resistance monitoring technique to determine the corrosivity of gaseous environments. The technique is based on the comparison of the electrical resistance of two legs of a bridge circuit which are exposed to the corrosive gases with the resistance of two legs of the circuit which are shielded from the corrosive gases by the presence of an inert overcoat. The circuit is formed from thin metal films such that corrosion of the film removes metal from the conductive path thus increasing the resistance. Modifications of this technique have been successfully used to monitor mixed flowing gas chamber corrosivity on a real time basis. Sensitivity of the resistance bridge can be adjusted by using thinner metal films such that modest corrosion films produce more significant resistance shifts.

NOTE X2.1—A limitation of the mass gain and resistance techniques is that the same observed rate of change can be accomplished by different gas concentrations in a multiple gas chamber. Use of multiple metals which are sensitive to different gases is required to assure control of gas ratios.

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