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Standard Guide for
Corrosion Tests in High Temperature or High Pressure Environment, or Both[1]

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1. Scope

1.1 This guide covers procedures, specimens, and equipment for conducting laboratory corrosion tests on metallic materials under conditions of high pressure (HP) or the combination of high temperature and high pressure (HTHP). See 3.2 for definitions of high pressure and temperature.

1.2 The procedures and methods in this guide are applicable for conducting mass loss corrosion, localized corrosion, and electrochemical tests as well as for use in environmentally induced cracking tests that need to be conducted under HP or HTHP conditions.

1.3 The primary purpose for this guide is to promote consistency of corrosion test results. Furthermore, this guide will aid in the comparison of corrosion data between laboratories or testing organizations that utilize different equipment.

1.4 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

1.5 *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 establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E8/E8M Test Methods for Tension Testing of Metallic Materials

G1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens

G4 Guide for Conducting Corrosion Tests in Field Applications

G5 Reference Test Method for Making Potentiodynamic Anodic Polarization Measurements

G30 Practice for Making and Using U-Bend Stress-Corrosion Test Specimens

G31 Guide for Laboratory Immersion Corrosion Testing of Metals

G34 Test Method for Exfoliation Corrosion Susceptibility in 2XXX and 7XXX Series Aluminum Alloys (EXCO Test)

G38 Practice for Making and Using C-Ring Stress-Corrosion Test Specimens

G39 Practice for Preparation and Use of Bent-Beam Stress-Corrosion Test Specimens

G46 Guide for Examination and Evaluation of Pitting Corrosion

G49 Practice for Preparation and Use of Direct Tension Stress-Corrosion Test Specimens

G59 Test Method for Conducting Potentiodynamic Polarization Resistance Measurements

G78 Guide for Crevice Corrosion Testing of Iron-Base and Nickel-Base Stainless Alloys in Seawater and Other Chloride-Containing Aqueous Environments

G106 Practice for Verification of Algorithm and Equipment for Electrochemical Impedance Measurements

G129 Practice for Slow Strain Rate Testing to Evaluate the Susceptibility of Metallic Materials to Environmentally Assisted Cracking

G170 Guide for Evaluating and Qualifying Oilfield and Refinery Corrosion Inhibitors in the Laboratory

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3. Terminology

3.1 *Definitions*—The definitions of terms given in Terminology G193 shall be considered as applying to this guide.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *high temperature and high pressure, HTHP, n*—for the purpose of this guide, any combination of pressure and temperature that requires the use of an autoclave.

3.2.2 *effectively non-reactive, adj*—free of significant mass loss or localized corrosion, stress corrosion cracking (SCC), or other embrittlement phenomena in the test environment; not contaminate the test environment with corrosion or other reaction products; and not significantly consume, absorb, or adsorb reactive chemical species from the test environment.

3.3 *Abbreviations and Acronyms:*

3.3.1 *AI*—artificial intelligence

3.3.2 *ALARP*—as low as reasonably practicable

3.3.3 *CUE*—corrosion under excursions

3.3.4 *HV*—high velocity

3.3.5 *ML*—machine learning

3.3.6 *PVT*—pressure volume temperature

3.3.7 *RT*—radiography testing (X-ray)

3.3.8 *SCC*—stressed-corrosion cracking

3.3.9 *SME*—subject matter expert

3.3.10 *UT*—ultrasonic testing

4. Summary of Guide

4.1 This guide describes the use of corrosion coupons, stress corrosion cracking specimens, and electrochemical electrodes in HP and HTHP environments. It also includes guidelines for the use of high-pressure test cells with these specimens to obtain reproducible, accurate corrosion test data.

4.2 Typically, HP and HTHP tests involve exposure of test specimens to a liquid (aqueous or non-aqueous), gaseous or multiphase environment, or both, in an appropriate test cell. The test cell shall be able to resist corrosion and cracking (mechanical and environmental) in the test environment while containing the pressurized, heated environment. Furthermore, the test specimens in the HP or HTHP test, or both, can be exposed in either stressed or unstressed condition in either the free corroding state or under electrochemical polarization.

5. Significance and Use

5.1 Autoclave tests are commonly used to evaluate the corrosion performance of metallic and non-metallic materials under simulated HP and HTHP service conditions. Examples of service environments in which HP and HTHP corrosion tests have been used include chemical processing, petroleum production and refining, food processing, pressurized cooling water, electric power systems, and aerospace propulsion.

5.2 For the applications of corrosion testing listed in 5.1, the service environment involves handling corrosive and potentially hazardous media under conditions of high pressure or high temperature, or both. The temperature and pressure, among other parameters, usually drive the composition and properties of the aqueous phase and, hence, the severity of the corrosion process. Consequently, the laboratory evaluation of corrosion severity cannot be performed in conventional low pressure glassware without making potentially invalid assumptions as to the potential effects of high temperature and pressure on corrosion severity.

5.3 Therefore, there is a substantial need to provide standardized methods by which corrosion testing can be performed under HP and HTHP. In many cases, however, the standards used for exposure of specimens in conventional low-pressure glassware experiments cannot be followed due to the limitations of access, volume, and visibility arising from the construction of high-pressure test cells. This guide refers to existing corrosion standards and practices, as applicable, and then goes further in areas in which specific guidelines for performing HP and HTHP corrosion testing are needed.

6. Apparatus

6.1 *Test Cell:*

6.1.1 Shall be constructed to applicable standards and codes so that it will have an adequate pressure rating to handle the test pressure safely.

6.1.2 Shall be made of materials that are corrosion resistant and effectively non-reactive with the test environment.

6.1.3 Shall have a seal mechanism that can withstand both the pressures, temperatures, and corrosive environment to be used in the test. Periodic hydrostatic testing of the test cell is recommended to ensure pressure retaining capabilities.

6.1.4 Shall be designed to have the necessary inlet and outlet ports to allow the test environment to be established in a controllable manner, monitored and sampled during the exposure period, released in a controlled manner at the completion of the test, and, if over temperature or pressure conditions may occur, adequate over pressure release and over temperature control equipment should be used.

6.2 *Test Cell Feedthroughs:*

6.2.1 Should be designed to minimize frictional forces when external loading fixtures are used for stressing specimens.

6.2.2 May be designed to balance the internal pressure in the test vessel with the external stressing assembly.

6.2.3 Shall be designed so that the electrodes or stressing rods and specimens cannot be ejected from the test cell under pressure.

6.2.4 Shall provide the electrical isolation of the specimen from the test cell unless galvanic coupling is specifically desired.

6.3 *Specimen Under Applied Load:*

6.3.1 Any frictional or pressure forces (or thermal expansion) acting on the specimen through the stressing fixtures should be taken into account when determining the actual load on the specimen.

6.3.2 Gripping devices should be designed such that they are in compliance with Test Methods E8/E8M where application of load to the specimen is required.

6.4 *Agitation:*

6.4.1 Agitation can be achieved by using a magnetic stir bar/plate, mechanically agitating the autoclave, for example, rollers within an oven recirculating the fluid through a pump, or using magnetic/mechanical feedthrough stirrers designed for this purpose. Care should be taken to ensure that components of the pump or stirrer are inert to the test environment.

NOTE 1—The potential for oxygen contamination and pressure loss increase with the use of pumps and additional autoclave ports.

6.4.2 Descriptions of test methods and equipment designs for testing under turbulent flow conditions, defined as a function of wall shear stress or mass transfer coefficient, can be found collectively in Guide G170, Practice G184 (rotating cage autoclaves), Practice G185 (rotating cylinder electrodes), and Practice G208 (jet impingement).

7. Reagents

7.1 In corrosion testing, providing a reproducible chemical environment in which to expose the corrosion test specimens is necessary.

7.2 In cases where the test environment is established by the mixing of chemicals in the laboratory, chemicals of reagent-grade purity with known contaminant levels are recommended.

7.3 In HP/HTHP corrosion testing, a common practice is to conduct tests in environments that have been sampled and retrieved from field or plant locations. Detailed information as to the chemical composition of the environment should be obtained. Particular attention should be given to the levels of impurities and contaminants that may be in the environment. Furthermore, under some conditions, these environments may be prone to changes after sampling or during testing, which can affect the corrosion test results.

7.4 In all cases, it is recommended that the test environment be fully documented with respect to its chemical composition.

8. Sampling and Test Specimens

8.1 *Sampling Selection Process*—Refer to G4, Standard Guide for Conducting Corrosion Tests in Field Applications.

8.2 *Preparation of Specimens:*

8.2.1 Frequently, the primary objective is to prepare a reproducible metallic surface with an absolute minimum of cold working followed by cleaning and degreasing. However, there are cases where the as-received surface is the desired test surface, or where the effect of cold working is to be studied.

8.2.2 Since test cells for HP and HTHP tests are usually of metallic construction, care must be taken to electrically isolate the specimens from the test cell unless galvanic coupling is specifically desired in the test. In cases where the test cell is used as a member of a galvanic couple, care must be taken to ensure that the galvanic action (anodic or cathodic) does not degrade the integrity of the test cell.

8.3 *Corrosion Specimens:*

8.3.1 Prepare specimens used in HP or HTHP corrosion tests in accordance with Practice G1 and Guide G31. Commonly, test cells used for HP and HTHP exposure tests are restricted in volume. The available volume in the test cell often decreases with increasing pressure rating. Therefore, it is frequently necessary to restrict the size and surface area of corrosion coupons used in HP and HTHP corrosion tests to attain a given volume to area ratio.

8.3.2 A minimum ratio, R, is often set for the volume of corrosive liquid to the surface area of the metal that can be corroded, for example, 200 L/m^2 to 400 L/m^2 or 20 mL/cm^2 to 40 mL/cm^2 (Guide G31, Test Method G34, and Practice G185). The origin of this concept is to minimize the buildup of corrosion products, which could subsequently impact solution pH, scaling tendencies, general corrosion rate, pit initiation, and pitting rates. Additionally, R can impact the rate at which reactants are depleted and potentially undesirable changes to the test environment occur. Consequently, an acceptable value for R will depend on the highest expected corrosion rate and the test duration. Another factor, not accounted for by R, arises from the possibility that certain chemicals, for example, components in a corrosion inhibitor, may adsorb (competitively) on metal surfaces other than the test specimen. Hence, in designing a test, one might also consider how the ratio of total immersed metal surface area to liquid volume compares with that in the field.

8.4 *Stressed Corrosion Specimens:*

8.4.1 Methods for the fabrication and use of appropriate stressed specimens are given in Section 2. These include tension, bent beam, C-ring, and U-bend specimens in accordance with Practices G129, G49, G39, G38, and G30, respectively. Fracture mechanics specimens can also be accommodated.

8.4.2 For similar reasons given in 8.3, when testing multiple specimens, it is recommended that the size of the specimens be restricted to the smallest applicable specimen provided for under the appropriate standards.

8.4.3 Because of the limited access of the specimens in HP and HTHP tests, self-stressed specimens are usually more convenient than specimens that require external stressing fixtures.

8.4.4 In cases such as direct tension and fracture mechanics tests, use of external loading frames and fixtures in conjunction with HP and HTHP corrosion tests may be desirable. In these cases, take both the frictional (sealing) forces and pressure forces acting on the specimens into account when determining the effect of applied stress.

8.5 *Electrochemical Electrodes:*

8.5.1 Prepare electrodes for use in HP and HTHP corrosion studies as described in Reference Test Method G5, Test Method G59, and Practice G106.

8.5.2 Cylindrical electrode specimens in which only the lower portion of the electrode is exposed to the liquid phase of test environment, and the electrical connections are made externally to the test cell are a convenient geometry. Care shall be taken to isolate the electrodes electrically from the test cell. Other electrode geometries and designs may be used that facilitate feedthrough and electrical isolation.

8.5.3 A critical portion of the HP or HTHP electrochemical system is the design and construction of the reference electrode. It is common to use external reference cells that use stable reference systems such as Ag/AgCl or another stable electrochemical reference system that can be enclosed in a separate pressure containing compartment. This cell is then connected to the test cell by means of a salt bridge and is pressure balanced with the test cell to minimize ingress of contaminants into either the test cell or the reference electrode. Alternatively, an inert or corroding metal electrode can be used as a non-standard reference electrode in some cases. Examples of such non-standard reference electrodes include platinum, graphite, or other metal with known stable corrosion potential. However, one problem that can occur with this technique is a drift in reference potential with time. Care should be taken when using such methods. These non-standard reference electrodes can effectively give a measure of relative potential even if the absolute potential is not known.

9. Test Environment

9.1 Choose the test environment to give the most accurate possible representation of the service environment (within the constraints of the equipment available) or provide a simple screening environment. In many cases, the test cells used to conduct HP tests are limited in volume and may not be designed to accommodate replenishment of the environment.

9.1.1 In the case of service environment simulation, monitoring concentrations of chemical species in the test cell may be needed to check that the environment is within a specified range of compositions (see 9.6). In some cases, replenishment of the test environment may be necessary. The need for replenishment may be influenced by factors such as test duration and the stability/life of any additives relative to the test duration.

9.1.2 In the case of simple screening environments, allowance for greater latitude in the variance of the test environment from service conditions is acceptable, and chemical monitoring may not be required.

9.2 *Test Temperature:*

9.2.1 Care should be taken to control the temperature as closely as possible because small temperature changes can significantly affect process rates. The test temperature should be controlled to ±3 °C of the targeted temperature. Many temperature controllers are susceptible to overshooting the set point upon initial temp-up. The time required to reach the test temperature should be recorded and kept constant throughout

the test matrix. If the set point is exceeded, the magnitude and duration of the deviation should be documented.

9.2.2 Temperature of the liquid phase can be measured in one location if the specimens are totally contained therein, and the solution is sufficiently agitated. However, for large test cells, temperature gradients higher than specified in 9.2.1 can exist ,and care should be taken to monitor the temperature close to the specimens using thermocouples contained in corrosion resistant sheaths. In this case, the thermocouples should be positioned to limit their impact on the fluid flow over adjacent coupons.

9.2.3 In tests in which the specimens are exposed to the gaseous or vapor phase, care shall be taken to obtain direct measurements of specimen temperature. When the test vessel is heated externally, the vessel temperature may greatly exceed the specimen temperature. Internally heating the specimen may be possible in the gaseous environment. Such a procedure is particularly useful when conditions of heat transfer are being simulated.

9.3 *Pressure:*

9.3.1 The pressure shall be monitored and recorded as required by internal/external safety protocols, and the project's scope of work using either a pressure gauge or pressure transducer.

9.3.2 Pressure sensors accuracy and range should be selected to measure changes precisely at least 1 % of the target pressures. Sensors should be recalibrated at regular intervals because drift can occur over time.

9.3.3 An acceptability criterion for the departure of pressure from target values and corrective actions should be defined depending on the corrosion system. Departures may occur during the test because of minor leaks or changes in the environment (for example, gas consumption, hydrogen generation).

9.3.4 Care shall be taken to select materials of construction properly for these measurement devices if exposed directly to the test environment. Methods to minimize corrosion of pressure monitoring equipment are to provide for an isolation valve between the monitoring equipment or use a diaphragm seal that transmits the pressure from the test cell to the monitoring equipment by means of a chemically inert media.

9.4 *Liquid Constituent(s):*

9.4.1 The volume of the liquid phase(s) should be recorded along with the autoclave's effective volume, that is, the volume of the autoclave minus the test fixtures and specimens. It is common for the liquid phase(s) to account for up to 80 % of the effective volume. Thermal expansion of the liquid phase or the creation of a supercritical fluid phase, or both, can result in a rapid and dangerous pressure increase. As noted in 9.5.2, thermodynamic models can be useful in predicting the magnitude of thermal expansion and phase changes.

9.4.2 Sometimes a second liquid phase may be present (for example, oil) or a liquified gas or supercritical phase may form at HTHP conditions. In such a case, this second liquid phase should be accounted for in the total liquid volume. When testing acid gases, the second liquid phase uptake of the corrodents may be significant, and simulation of the environment is even more relevant.

9.5 *Gaseous Constituent(s):*

9.5.1 Gases can be used separately as a test environment or in combination with liquid environments to provide aeration, deaeration, saturation of soluble gases, and pressurization.

9.5.2 Care should be taken to add the correct amount of each gaseous component to a closed batch system. PVT calculations based on equations of state with an electrolyte model and accounting for autoclave dead volumes, thermal expansion, phase changes, solubility, nonideality, and their dependence on pressure and temperature can be helpful in this regard. When testing corrosion produced by acid gases (for example, CO_2, H_2S), there are three modeling options available to attempt to reproduce a real environment under laboratory conditions: (*1*) corrosive gas partial pressure equivalence, (*2*) corrosive gas fugacity equivalence (**1-5**),[3] and (*3*) corrosive gas in liquid concentration equivalence (**6-12**). The assumption of ideality implicit in option (*1*) tends to become less tenable as conditions become more extreme. Different factors may prompt selection of options (*2*) or (*3*). If the selection is not clear, it may be prudent to calculate the gas loadings by both methods and to analyze the difference in the context of inherent approximations in the models. If, in this case, the models lead to significantly different charges, it may be worthwhile to run separate laboratory tests with values calculated using both models to ensure that a conclusion is not reached that hinges on an uncertainty or assumption.

9.5.3 If a second liquid phase is present or can be formed at HTHP conditions, it is important to be aware of the limitations of thermodynamic models. If oil is being used, limitations can include, but are not limited to: (*1*) representation of oil composition and properties, and (*2*) partitioning of components (including both corrosive and inert gases) across the phases present. If any of the gaseous constituents can become supercritical, there may be additional limitations, for example, (*1*) identification of the critical point, (*2*) representation of supercritical phase properties, and (*3*) partitioning of components across the phases present.

9.5.4 There are advantages and disadvantages to different gas-loading strategies. On the one hand, loading at room temperature can lead to pessimistic results, for example, because of: (*1*) H_2S overloading from high initial adsorption on vessel walls; (*2*) CO_2 supersaturation from high initial solubility combined with finite equilibration times; and (*3*) to account for high acid gas solubilities at room temperature. On the other hand, loading at the test temperature will require higher pressures from gas cylinders and, in special cases, a booster compressor. Best practices for gas loading should be used to maintain safety, avoid leaks, and obtain a representative environment. For example, precautions are needed if a liquidized gas source will be used because volatilization in the autoclave can lead to a rapid pressure increase; PVT calculations should demonstrate clearly that such additions would not exceed the pressure rating for the equipment.

9.5.5 In some cases, it is necessary to add liquefied gas constituents at room temperature that then convert to a pres-

surized gas upon heating to elevated temperature. In this case, the quantity of the liquefied gas that is added can be controlled by weight or monitoring the increase in autoclave pressure.

9.5.6 If deaerated conditions are required, care should be taken to remove air from the test vessel and the liquid constituents adequately. Cyclic vacuuming and inert gas purging of the test vessel are often used for this purpose. If a vacuum will be used for deaeration, it is important to: (*1*) consider the use of vacuum-compatible rupture disks, and (*2*) ensure that the system will hold the vacuum, that is, will not allow the ingress of oxygen. The latter can be evaluated by sealing the system under vacuum and monitoring any changes in total pressure with time.

9.5.7 For tests that aim to exclude oxygen, it is advantageous to check that the oxygen level is sufficiently low in autoclaves and preparation vessels at the start of a test. "Sufficiently low" in the vapor is dictated by the corresponding level that would exist in the corrosive medium at equilibrium. For aqueous corrosion, it has been shown that the liquid-phase O_2 concentration should be held below 5 ppb and preferably below 1 ppb (Practice G185) to avoid oxygen corrosion effects. Application of Henry's law (or an equivalent solubility equation) allows the corresponding level in the gas phase to be estimated. In the vapor phase, 10 ppm O_2 is often selected on this basis, and it corresponds to a low detection limit for some oxygen analyzers. One might advocate for oxygen measurements during a test, but this would incur hazards involved with sampling from HTHP equipment (see 9.6), and the data would not be able to assess oxygen that had already reacted.

9.5.8 Where reactive constituents are present in the test gas, it may be necessary to allow for sufficient vapor space to act as a reservoir of the reactive species or replenish the gas in the test cell on either a periodic or continuous basis. If this condition is suspected, detailed chemical analysis of the test environment versus time may be required. This procedure will allow for the frequency of replenishment to be determined. The magnitude of an acceptable percent change in chemical composition will depend on the chemicals, the test conditions, the anticipated impact of a change from the original value, and the goal of the test.

9.6 *Test Monitoring*—It might be possible to track depletion or buildup of chemical species approximately through indirect quantities such as total pressure or in-situ pH if measurable. Subsampling of fluids during a test may be problematic because of safety concerns removing hot fluids from a pressurized vessel as well as changes imparted to the remaining fluids. Moreover, it may be difficult to capture a representative subsample. If continuous or periodic replenishment of some or all the fluids is conducted, the fluids removed can be analyzed. Otherwise, samples taken after a test has ended can be taken and analyzed. In this case, tests of different durations could be used to obtain information on concentration changes with time.

10. Procedure

10.1 Clean, weigh, and assemble the test specimens on suitable fixtures with electrical isolation. Care should be taken to secure the specimens and position them in the test cell, as

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