

Standard Test Method

Laboratory Testing of Metals for Resistance to Sulfide Stress Cracking and Stress Corrosion Cracking in H₂S Environments

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Foreword

This standard addresses the testing of metals for resistance to cracking failure under the combined action of tensile stress and corrosion in aqueous environments containing hydrogen sulfide (H_2S). This phenomenon is generally termed sulfide stress cracking (SSC) when operating at room temperature and stress corrosion cracking (SCC) when operating at higher temperatures. In recognition of the variation with temperature and with different materials this phenomenon is herein called environmental cracking (EC). For the purposes of this standard, EC includes only SSC, SCC, and hydrogen stress cracking (HSC).

The primary purpose of this standard is to facilitate conformity in testing so that data from different sources can be compared on a common basis. Consequently, this standard aids the evaluation and selection of all types of metals and alloys, regardless of their form or application, for service in H_2S environments. This standard contains methods for testing metals using tensile, bent-beam, C-ring, and double-cantilever-beam (DCB) test specimens. Certain ASTM⁽¹⁾ standard test methods have been listed as references for supplementary tests, creating a comprehensive test method standard. In addition, the four-point bent-beam test method is also referenced as a supplementary test.^{1,2} This standard is intended for use by laboratory and materials personnel to facilitate conformity in testing.

SSC of metals exposed to oilfield environments containing H_2S was recognized as a materials failure problem by 1952. Laboratory data and field experience have demonstrated that even extremely low concentrations of H_2S may be sufficient to lead to SSC failure of susceptible materials. In some cases H_2S can act synergistically with chlorides to produce corrosion and cracking (SSC and other mode) failures. However, laboratory and operating experiences have also indicated to materials engineers the optimum selection and specification of materials having minimum susceptibility to SSC. This standard covers test methods for SSC (at room temperature) and SCC (at elevated temperature), but other failure modes (e.g., hydrogen blistering, hydrogen-induced cracking [HIC], chloride stress corrosion cracking [SCC], pitting corrosion, and mass-loss corrosion) must also be considered when selecting materials for use in sour (H_2S -containing) environments.

The need for better understanding of the variables involved in EC of metals in oilfield environments and better correlation of data has become apparent for several reasons. New design requirements by the oil and gas production industries call for higher-strength materials that, in general, are more susceptible to EC than lower-strength alloys. These design requirements have resulted in extensive development programs to obtain more resistant alloys and/or better heat treatments. At the same time, users in the petroleum refining and synthetic fuels industries are pushing present materials much closer to their mechanical limits.

Room-temperature (SSC) failures in some alloys generally are believed to result from hydrogen embrittlement (HE). When hydrogen is cathodically evolved on the surface of a metal (as by corrosion or cathodic charging), the presence of H_2S (and other compounds, such as those containing cyanides and arsenic) tends to cause hydrogen atoms to enter the metal rather than to form hydrogen molecules that cannot enter the metal. In the metal, hydrogen atoms diffuse to regions of high triaxial tensile stress or to some microstructural configurations where they become trapped and decrease the ductility of the metal. Although there are several kinds of cracking damage that can occur in metals, delayed brittle fracture of metals resulting from the combined action of corrosion in an aqueous sulfide environment and tensile stresses (failure may occur at stresses far below the yield stress) is the phenomenon known as SSC.

In some cases, however, failure may be the result of localized anodic corrosion processes that may or may not involve hydrogen. In such instances failure is the result of anodic stress corrosion cracking (SCC). Such failures have historically been termed SSC even though their cause may not be hydrogen.

⁽¹⁾ ASTM International (ASTM), 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959.

This standard was originally published in 1977 by NACE International Task Group T-1F-9, a component of Unit Committee T-1F on Metallurgy of Oilfield Equipment. The standard was revised in 1986, 1990, and 1996 by Task Group T-1F-9. It was revised in 2005 by Task Group (TG) 085 on Sulfide Corrosion Cracking: Metallic Materials Testing Techniques. TG 085 is administered by Specific Technology Group (STG) 32 on Oil and Gas Production—Metallurgy and is sponsored by STG 62 on Corrosion Monitoring and Measurement—Science and Engineering Applications. The standard is issued by NACE under the auspices of STG 32.

In NACE standards, the terms *shall*, *must*, *should*, and *may* are used in accordance with the definitions of these terms in the *NACE Publications Style Manual*, 4th ed., Paragraph 7.4.1.9. *Shall* and *must* are used to state mandatory requirements. The term *should* is used to state something considered good and is recommended but is not mandatory. The term *may* is used to state something considered optional.

NACE International Standard Test Method

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Section 1: General

1.1 This standard covers the testing of metals subjected to tensile stresses for resistance to cracking failure in low-pH aqueous environments containing H₂S. Carbon and low-alloy steels are commonly tested for EC resistance at room temperature where SSC susceptibility is typically high. For other types of alloys the correlation of EC susceptibility with temperature is more complicated.

1.2 This standard describes the reagents, test specimens, and equipment to use, discusses base material and test specimen properties, and specifies the test procedures to follow. This standard describes four test methods:

Method A—Standard Tensile Test

Method B—Standard Bent-Beam Test

Method C—Standard C-Ring Test

Method D—Standard Double-Cantilever-Beam (DCB) Test

Sections 1 through 7 of this standard give general comments that apply to all four test methods. Sections 8 through 11 indicate the test method to follow for each type of test specimen. General guidelines to help to determine the aptness of each test method are given at the beginning of each test method description (Sections 8 through 11). Reporting of the test results is also discussed.

1.3 Metals can be tested for resistance to EC at temperatures and pressures that are either ambient (atmospheric) or elevated.

1.3.1 For testing at ambient conditions, the test procedures can be summarized as follows: Stressed test specimens are immersed in acidified aqueous environments containing H₂S. Applied loads at convenient increments can be used to obtain EC data.

1.3.2 For testing at temperatures higher than 27°C (80°F), at either atmospheric or elevated pressure, Section 7 describes an alternative test technique. All methods (A, B, C, and D) are adaptable to this technique.

1.4 This standard may be used for release or acceptance testing to ensure that the product meets a certain minimum level of EC resistance as prescribed in API⁽²⁾ Specification 5CT,³ ISO⁽³⁾ 11960,⁴ or as prescribed by the user or purchaser. This standard may also provide a quantitative measure of the product's EC resistance for research or informational purposes. This rating may be based on:

- Method A The highest no-failure stress in 720 hours.
- Method B The statistically based critical stress factor (S_c) for a 50% probability of failure in 720 hours.
- Method C The highest no-failure stress in 720 hours.
- Method D The average K_{ISSC} (threshold stress intensity factor for SSC) for valid tests of replicate test specimens.

1.5 Safety Precautions: H₂S is an extremely toxic gas that must be handled with care. (See Appendix A.)

Section 2: EC Testing Variability

2.1 Interpretation of stress corrosion test results is a difficult task. The test methods contained in this standard are severe, with accelerated tests making the evaluation of the data extremely difficult. In testing the reproducibility of the test methods among different laboratories, several undesirable side effects (frequent with many accelerated tests) that must be noted include:

2.1.1 The test environment may cause failure by HIC and hydrogen blistering. This is especially true for lower-strength steels not usually subject to SSC. HIC may be detected by visual and metallographic observations. Blistering is normally visible on the test specimen surface. (For further information regarding this phenomenon, see NACE Standard TM0284.⁵)

2.1.2 The test environment may corrode some alloys that normally do not corrode in actual field service and thereby induce EC failures in alloys that ordinarily do not fail by EC. This problem is especially acute with the martensitic and precipitation-hardened stainless steels.

2.2 Furthermore, other aspects to be considered in the selection of test method(s) include:

2.2.1 Material anisotropy affecting mechanical properties and EC susceptibility can be an important parameter. The fracture path in the test specimen should match what is anticipated in the actual component.

⁽²⁾ American Petroleum Institute (API), 1220 L St. NW, Washington, DC 20005.

⁽³⁾ International Organization for Standardization (ISO), 1 rue de Varembé, Case postale 56, CH-1211 Geneva 20, Switzerland.

2.2.2 Galvanic effects between dissimilar metals can either accelerate or suppress cracking susceptibility. Examples of this behavior are accelerated EC in some nickel-based corrosion-resistant alloys (CRAs) and reduced EC in some duplex stainless steels when these materials are coupled to electrochemically less-noble materials such as carbon and low-alloy steels.

2.2.3 Test temperature affects cracking susceptibility. Test temperatures above 24°C (75°F) can reduce SSC severity in steels, whereas test temperatures below 24°C (75°F) can increase SSC severity.

2.2.4 Different test methods may not necessarily provide the same rankings of like materials.

2.2.5 Material inhomogeneity, such as weldments and segregation, can affect test results. This is particularly true when comparing results from tests that evaluate a large volume of material (tensile test) versus a small volume of material (bent-beam test).

2.2.6 Maximum no-failure stresses for a specified exposure period should be considered apparent threshold stresses. Longer exposure times or larger numbers of test specimens may result in lower threshold values.

2.2.7 EC test results can show statistical variability. Replicate testing may be needed to obtain a representative value characterizing resistance to EC.

2.2.8 Some test specimens are better suited than others for measuring EC resistance in localized areas (e.g., near surfaces or other features, and in weld zones).

2.2.9 Some types of EC tests require considerably more time than others for determination of EC resistance.

Section 3: Reagents

3.1 Reagent Purity

3.1.1 The test gases, sodium chloride (NaCl), acetic acid (CH₃COOH), sodium acetate (CH₃COONa), and solvents shall be reagent grade or chemically pure (99.5% minimum purity) chemicals. (See Appendix B.)

3.1.2 The test water shall be distilled or deionized and of quality equal to or greater than ASTM Type IV (ASTM D 1193⁶). Tap water shall not be used.

3.2 Inert gas shall be used for removal of oxygen. Inert gas shall mean high-purity nitrogen, argon, or other suitable nonreactive gas.

Section 4: Test Specimens and Material Properties

4.1 The location and orientation of the test specimens to be removed from the product should be carefully considered so that test results provide the most meaningful indication of the performance to be expected from that product when placed in service. All test specimens in a set should be taken from metallurgically equivalent positions (i.e., all test specimens should have the same orientation with similar or nearly the same microstructure and mechanical properties).

4.2 Tensile testing in accordance with standard test methods such as ASTM A 370⁷ shall be used to determine base material properties. Two or more test specimens shall be pulled, and the individual test results shall be averaged to determine the yield and ultimate strengths, percent elongation, and percent reduction in area for the material. Machining a tensile test specimen from material adjacent to and in the same position and orientation as the EC test

specimen to be tested can minimize material property variations that normally occur from test specimen to test specimen.

4.3 A number of fundamental material properties correlate with EC susceptibility. Consequently, all pertinent data on chemical composition, mechanical properties, heat treatment, and mechanical histories (such as percent cold reduction or prestrain) shall be determined and reported with the tensile test data. Each different heat treatment and microstructure of a material of a fixed chemical composition shall be tested as though it were a different material.

4.4 Hardness may be measured on the test specimen before or after exposure to the test environment. However, these measurements shall not be made on the stressed evaluation portion of the test specimen.

Section 5: Test Vessels and Fixtures

5.1 The size, shape, and entry ports of the test vessel shall be determined by the actual test specimens and test fixtures used to stress the test specimens.

5.2 Test vessels shall be capable of being purged to remove oxygen before beginning the test and of keeping air out during the test. Using a small outlet trap on the H₂S effluent line to maintain 250 Pa (1.0 in H₂O) back pressure on the test vessel prevents oxygen entry through small leaks or by diffusion up the vent line. (See Appendix B, section titled "Reasons for Exclusion of Oxygen.")

5.3 Test vessels shall be sized to maintain the test solution volume within the specified limits relative to the test specimen surface area to standardize the drift of pH with time. (See each test method for specified limits.)

5.4 Test vessels shall be constructed from materials that are inert to the test environment. While some plastic test vessels give satisfactory service, others may cause varying test results from the time they are new until after they have been in continuous use. Glass test vessels have not exhibited this tendency.

5.5 Test specimens shall be electrically isolated from test

vessels and test fixtures made from dissimilar metals if the dissimilar metal is in contact with the test environment.

5.6 Rigid electrical insulating materials not exhibiting relaxation or flow under load should be selected for loading or deflecting the test specimen.

5.7 Galvanic Coupling

5.7.1 It may be necessary to evaluate the effects of galvanic coupling on EC resistance, such as in the case of coupling stainless alloys or CRAs to steel (see Paragraph 2.2.2).

5.7.1.1 To evaluate this, galvanic couples of iron or steel having a surface area between 0.5 and 1 times the exposed area of the test specimen should be bolted securely to the test specimen.

5.7.2 Particles of iron sulfide can be electrically conductive. If deposited on insulating materials, they can provide electrical connection between materials and affect the results of the tests.

Section 6: Test Solutions

6.1 Test Solution A

6.1.1 Test Solution A is an acidified and buffered, H₂S-saturated aqueous brine solution.

6.1.2 Test Solution A shall consist of 5.0 wt% sodium chloride and 0.5 wt% glacial acetic acid dissolved in distilled or deionized water (e.g., 50.0 g of NaCl and 5.0 g of CH₃COOH dissolved in 945 g of distilled or deionized water).

6.1.3 Test solution pH before or after H₂S saturation but before contact with a test specimen is expected to range between 2.6 and 2.8. Each laboratory shall have a demonstrated and documented procedure for purging to verify that the pH has not exceeded 3.0 for Test Solution A after purging. During the test, pH may increase but shall not exceed 4.0. If the pH exceeds 4.0, the test is invalid. If the test-solution-volume to test-specimen-surface-area ratio is maintained and steps are taken to exclude oxygen from the test vessel as specified in this standard, the pH will not exceed this value.

6.1.4 Test Solution A shall be used in Methods A, C, and D unless the use of Test Solution B or Test Solution C is specified by the user or purchaser.

6.2 Test Solution B

6.2.1 Test Solution B is an acidified and buffered, H₂S-saturated aqueous brine solution.

6.2.2 Test Solution B shall consist of 5.0 wt% sodium chloride, 2.5 wt% glacial acetic acid, and 0.41 wt% sodium acetate dissolved in distilled or deionized water (e.g., 50.0 g of NaCl, 25 g of CH₃COOH, and 4.1 g of CH₃COONa dissolved in 921 g of distilled or deionized water).

6.2.3 Test solution pH before or after H₂S saturation but before contact with a test specimen is expected to range between 3.4 and 3.6. During the test, pH may increase but shall not exceed 4.0. If the pH exceeds 4.0, the test is invalid. If the test-solution-volume to test-specimen-surface-area ratio is maintained and steps are taken to exclude oxygen from the test vessel as specified in this standard, the pH will not exceed this value.

6.2.4 Test Solution B is intended for use with carbon and low-alloy steels.

6.2.5 Test Solution B is allowed in Methods A, C, and D.

6.3 Test Solution C

6.3.1 Test Solution C is a buffered aqueous brine solution with a chloride content, H_2S partial pressure, and pH specified by the user or purchaser to simulate the intended service environment.

6.3.2 Test Solution C shall consist of distilled or deionized water containing 0.4 g/L sodium acetate (5 mM CH_3COONa) and chloride (added as $NaCl$) at the same concentration as the intended service environment.

6.3.3 Hydrochloric acid (HCl) or sodium hydroxide ($NaOH$) shall be added to achieve the specified pH. The test solution pH shall be measured at the start of the test and at the end of the test. The test solution pH shall be maintained within 0.2 pH units of the specified pH.

6.3.4 Test gas shall consist of a mixture of H_2S and carbon dioxide (CO_2), with H_2S content sufficient to produce the specified H_2S partial pressure of the intended service environment. The test gas shall be continuously bubbled through the test solution. The gas bubbling rate shall be optimized to maintain saturation of the test solution.

6.3.5 Test Solution C is intended for use with martensitic stainless steels.

6.3.6 Test Solution C is allowed in Methods A, C, and D.

Note: The combination of a lower acetate concentration (0.4 g/L) and acidification with HCl rather than acetic acid leads to a significantly reduced concentration of acetic acid in Test Solution C when compared with Test Solution B. Although this may be adequate for maintaining the bulk solution pH constant when testing corrosion-resistant alloys, the ability to resist an increase in pH at the metal-solution interface by dissociation of the acetic acid is lowered. As the tests are conducted with little convective flow, it is possible that the pH at the surface increases slightly, and for conditions on the borderline between active and passive behavior, this may lead to passivity, giving a nonconservative result. Bulk solution pH measurements do not necessarily indicate any limitation in the buffer capacity as the pH change is highly localized.

6.4 The test solution required for use in Method B is specified in Paragraph 9.3.

6.5 All reagents added to the test solutions shall be measured to $\pm 1.0\%$ of the quantities specified for the specific test solution.

6.6 The test solution shall be maintained at $24 \pm 3^\circ C$ ($75 \pm 5^\circ F$), except for Method D (the DCB test) in which the test temperature range is $24 \pm 1.7^\circ C$ ($75 \pm 3.0^\circ F$). Also, the test temperature range shall be specified in accordance with testing at elevated temperature (see Section 7). Any variations beyond this range shall be reported.

6.7 The test solution used for each test method shall be indicated on the material testing report.

Section 7: Testing at Elevated Temperature/Pressure

7.1 The dominant cracking mechanisms for most classes of materials in the presence of H_2S vary with temperature. Ferritic steels and ferritic and martensitic stainless steels crack primarily by a hydrogen (i.e., cathodic) mechanism and have maximum susceptibility near room temperature. For austenitic stainless steels, as temperature increases, cracking susceptibility increases due to the major contribution from anodic processes. Duplex stainless steels exhibit mixed behavior, with maximum susceptibility to cracking in a mid-range of temperatures. To facilitate testing in simulated service conditions or to predict worst-case conditions, and to facilitate testing with H_2S partial pressure exceeding 100 kPa (absolute) (14.5 psia), the following modified techniques are available.

7.2 Testing at elevated temperatures and pressures involves additional safety considerations compared with room temperature and atmospheric pressure testing. While some general guidance is given herein, it may not address all aspects and should be supplemented to accord with local safety requirements. Because H_2S may be consumed during the test, gas replenishment and continuous gas

bubbling techniques are described. The H_2S loss rate and its effect on the corrosiveness of the test environment are functions of several factors, including the corrosion rate of the test material and the partial pressure of H_2S in the test environment. Guidance is given on measures that experience has shown to be appropriate for maintaining the required H_2S partial pressure, but in all cases it is necessary to demonstrate, by measuring H_2S concentration in either the test solution or gas phase, that the required test conditions have been maintained. This information must be reported with the test data.

7.3 Test Equipment

The test equipment shall consist of a test vessel and accessory equipment rated to withstand corrosion and pressure commensurate with the test conditions and with an appropriate safety margin.

7.3.1 The test vessel shall be equipped with a thermocouple well or other means of measuring the temperature of the test solution, inlet and outlet ports

for gas, a dip tube on the inlet port, and a pressure-measuring device.

7.3.2 If continuous gas bubbling is to be used, a condenser on the outlet port may be used to limit loss of test solution. This has been found to be useful at temperatures greater than 50°C (120°F) and/or when the volume of the test solution is less than 200 mL.

7.3.3 A bursting (rupture) disc or pressure-relief valve is generally used for safety reasons.

7.3.4 The pressure-measuring device shall have an accuracy of $\pm 1\%$ of the maximum system pressure. If the pressure is measured by a gauge, the maximum system pressure shall be greater than 20% and less than 80% of gauge full scale. Schematic arrangements of test equipment used for the various test methods are shown in Figures 1 and 2.

7.3.5 Elastomeric seal materials, if used, must resist H_2S at the temperature of use as verified by independent measurement.

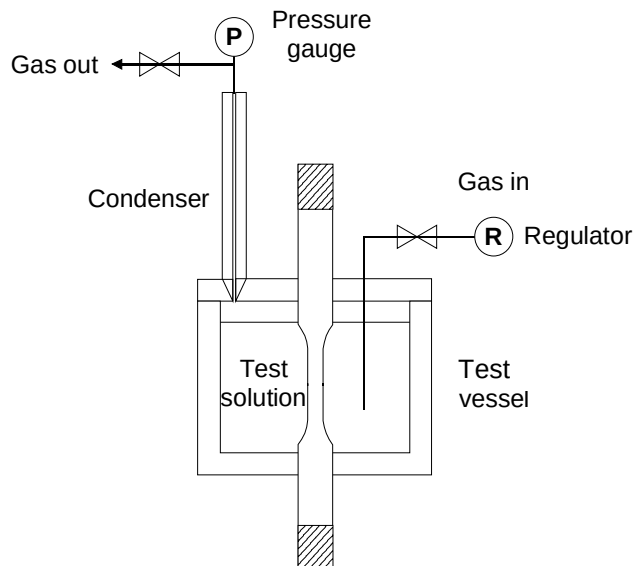


FIGURE 1: Schematic Arrangement of Test Equipment for Method A—NACE Standard Tensile Test

7.4 Test Solution

The test solution used in the test may be selected as required by the test specification. The test solution usually consists of brine (NaCl) at concentrations up to saturation. Buffered acidification is permitted, analogous to room-temperature methods. Also, the test solution may contain elemental sulfur.^{8,9,10}

7.5 Test Gas

The test gas is usually a mixture of two or more of the following: H_2S , CO_2 , and inert gas such as N_2 or Ar. At low H_2S partial pressures, tests in inert gas without CO_2 require careful interpretation because of corrosion product solubility effects.¹¹ The test gas mixture should be contained in a standard gas bottle equipped with a suitable pressure regulator (usually stainless steel) capable of gas delivery to the total test pressure required. A commercially supplied gas mixture with composition determined by analysis should be used.

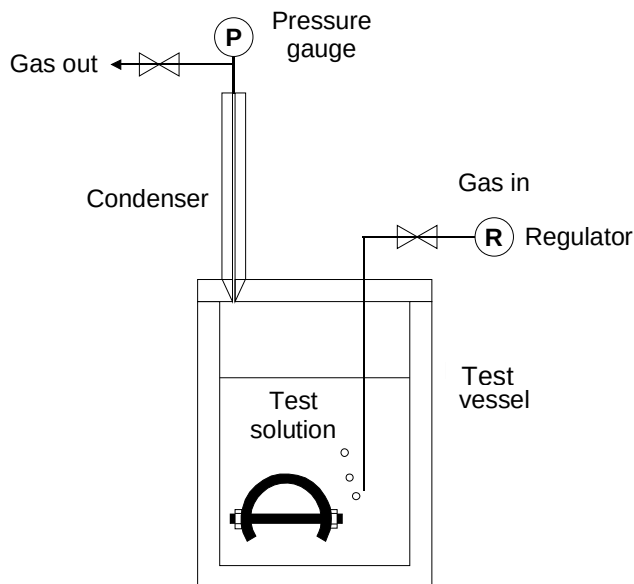


FIGURE 2: Schematic Arrangement of Test Equipment for Method B—NACE Standard Bent-Beam Test, Method C—NACE Standard C-Ring Test, and Method D—NACE Standard Double-Cantilever-Beam Test

7.6 Test Procedure

Test procedures shall be identical to those specified for room-temperature tests unless excepted or amended as follows:

7.6.1 The test solution and test specimen(s) shall be placed in the test vessel, then the test vessel shall be sealed and leak tested. Test vessels are usually tested for leaks with inert gas at 1.5 times the maximum test pressure.

7.6.2 The expansion of test solution on heating can fill the test vessel and risk explosion. The volume of test solution should be less than 75% of the total volume of the test vessel. Moreover, a greater safety margin (smaller percentage of total volume) should be used at temperatures exceeding 225°C (435°F).

7.6.3 The test solution shall be deaerated by bubbling inert gas through the gas inlet tube into the test solution for a minimum period of 1 h/L of test solution.

7.6.4 The H_2S partial pressure, p_{H_2S} , in the test environment shall be determined by one of the following two methods:

7.6.4.1 Test vessel heated before test gas admitted

7.6.4.1.1 The test vessel shall be heated with valves closed to test temperature and stabilized. System pressure (the vapor pressure of the test solution), P_1 , shall be measured.

7.6.4.1.2 Test gas shall be admitted to the vessel until the test pressure, P_T , is reached.

7.6.4.1.3 The H_2S partial pressure, p_{H_2S} , in the test environment is given approximately in Equation (1):

$$p_{H_2S} = (P_T - P_1) X_{H_2S} \quad (1)$$

where:

P_T = total absolute test pressure;
 P_1 = vapor pressure above the test solution;
 and
 X_{H_2S} = mole fraction of H_2S in the test gas.

7.6.4.2 Test gas admitted before test vessel heated

Test gas may be admitted to the test vessel before heating if a proven means of calculating p_{H_2S} can be demonstrated.

7.6.5 Test gas shall be replenished as needed to maintain the required test conditions (primarily H_2S partial pressure) as outlined in Paragraph 7.2. Continuous test gas bubbling at 0.5 to 1.0 mL/min or periodic test gas replenishment once or twice weekly has been found necessary when testing CRAs at H_2S partial pressures below 2 kPa (absolute) (0.3 psia) or carbon and alloy steels at H_2S partial pressures below 100 kPa (absolute) (14.5 psia). Test solution loss and ingress of oxygen during test gas replenishment shall be avoided.

7.6.6 The test duration shall be as specified for the applicable test method (A, B, C, or D). The test temperature for Methods A, B, and C shall be maintained within $\pm 3^{\circ}\text{C}$ ($\pm 5^{\circ}\text{F}$) of the specified test temperature and recorded manually on a daily basis or at shorter intervals by data recorder. For Method D, test temperature shall be maintained within $\pm 1.7^{\circ}\text{C}$ ($\pm 3.0^{\circ}\text{F}$). Pressure shall be monitored and recorded

daily. If test pressure falls by more than 40 kPa (6 psi) below the required test pressure, the test gas must be replenished.

7.6.7 At the test completion, the test vessel should be purged with inert gas while cooling to ambient temperature before opening. The load should be relaxed before cooling, if possible, when using equipment with external loading.

Section 8: Method A—NACE Standard Tensile Test

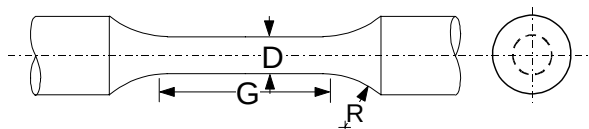
8.1 Method A, the NACE Standard Tensile Test, provides for evaluating metals for EC resistance under uniaxial tensile loading. It offers a simple unnotched test specimen with a well-defined stress state. EC susceptibility with Method A is usually determined by time-to-failure. Tensile test specimens loaded to a particular stress level give a failure/no-failure test result. When multiple test specimens are tested at varying stress levels, an apparent threshold stress for EC can be obtained.¹²

8.1.1 This section sets forth the procedure for testing at room temperature and atmospheric pressure. Special considerations for testing at elevated temperature and pressure are set forth in Section 7.

8.2 Test Specimen

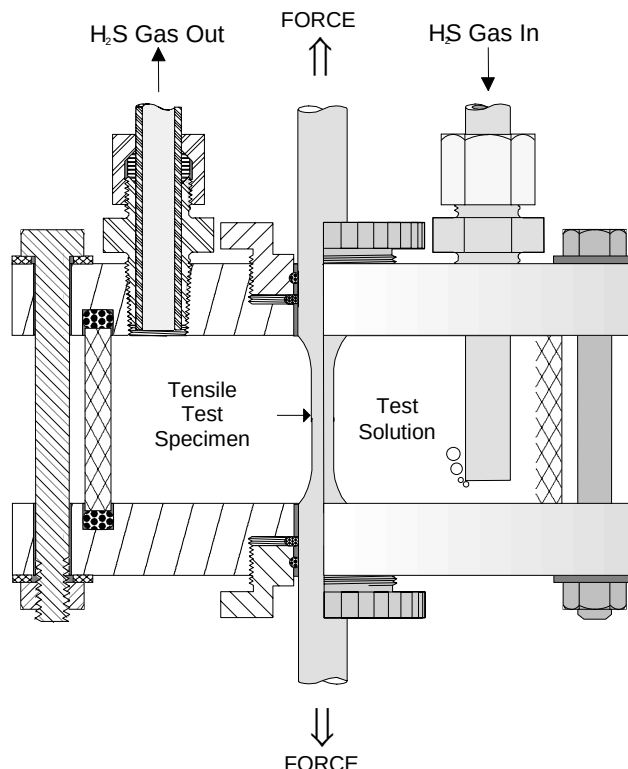
8.2.1 The size and shape of the material available for testing often restricts selection of test specimens. The orientation of the test specimen can affect the results and should be noted.

8.2.2 The gauge section of the standard tensile test specimen (see Figure 3[a]) shall be 6.35 ± 0.13 mm (0.250 ± 0.005 in.) in diameter by 25.4 mm (1.00 in.) long (see ASTM A 370). A subsize tensile test specimen with gauge section of 3.81 ± 0.05 mm (0.150 ± 0.002 in.) in diameter by 15 mm (0.60 in.) long is acceptable. After machining, tensile test specimens should be stored in a low-humidity area, in a desiccator, or in uninhibited oil until ready for testing.



Dimension	Standard tensile test specimen	Subsize tensile test specimen
D	6.35 ± 0.13 mm (0.250 ± 0.005 in.)	3.81 ± 0.05 mm (0.150 ± 0.002 in.)
G	25.4 mm (1.00 in.)	15 mm (0.60 in.)
R (min.)	15 mm (0.60 in.)	15 mm (0.60 in.)

(a) Dimensions of the tensile test specimens



(b) Tensile test specimen in an environmental chamber

FIGURE 3: Tensile Test Specimens

8.2.3 The radius of curvature at the ends of the gauge section shall be at least 15 mm (0.60 in.) to minimize stress concentrations and fillet failures.

8.2.3.1 Additional methods that have been found helpful in reducing fillet failures are to:

- (1) eliminate undercutting of fillet radii in machined test specimens; and
- (2) machine the test specimen gauge section with a slight (0.05- to 0.13-mm [0.002- to 0.005-in.]) taper that produces a minimum cross-section in the middle of the gauge section.

8.2.4 The ends of the test specimen must be long enough to accommodate seals for the test vessel and to make connections to the stressing fixture. (See Figure 3[b].)

8.2.5 The test specimen must be machined or ground carefully to avoid overheating and cold working in the gauge section. In machining operations, the final two passes should remove no more than a total of 0.05 mm (0.002 in.) of material. Grinding is also acceptable if the grinding process does not harden the material.

8.2.6 For all materials the final surface finish shall be 0.81 μm (32 $\mu\text{in.}$) or finer. Final surface finish may be obtained by mechanical polishing or electropolishing if the roughness requirement is met. Using any finishing process other than grinding must be reported with the test data. When electropolishing, bath conditions must be such that the test specimen does not absorb hydrogen during the procedure.

8.2.7 When the standard tensile test specimen cannot be obtained from the material because of its size or shape, an appropriate subsize tensile test specimen may be used. However, subsize tensile test specimens can produce shorter failure times than those observed for standard tensile test specimens. The report of test data using subsize tensile test specimens shall clearly state the use and size of subsize test specimens.

8.2.8 Test Specimen Identification

8.2.8.1 Stamping or vibratory stenciling may be used on the ends of the test specimen but shall not be used in the gauge section.

8.2.9 Test Specimen Cleaning

8.2.9.1 Before testing, test specimens shall be degreased with solvent and rinsed with acetone.

8.2.9.2 The gauge section of the test specimen shall not be handled or contaminated after cleaning.

8.3 Test Solutions (for Method A)—see Section 6.

8.4 Test Equipment

8.4.1 Many types of stress fixtures and test vessels used for stress corrosion testing are acceptable for Method A. Consequently, the following discussion emphasizes the test equipment characteristics required for selecting suitable items and procedures.

8.4.2 Tensile tests should be performed with constant-load devices or sustained-load (proof ring or spring-loaded) devices (see ASTM G 49¹³).

8.4.2.1 All loading devices shall be calibrated to ensure accurate application of load to the test specimen. The error for loads within the

calibration range of the loading device shall not exceed 1.0% of the calibration load.

8.4.2.2 The loading device shall be constructed to avoid torsional loads.

8.4.3 When susceptible materials are tested using sustained-load devices, it is possible for cracks to initiate and propagate only partially, not fully, through the test specimen (see Paragraph 8.7). Consequently, susceptibility determination from sustained-load test results requires the visual examination of the test specimens for the presence of part-through cracks. The determination may be difficult if the cracks are small and sparse or if obscured by corrosion deposits. However, testing with constant-load devices ensures that susceptible materials will separate completely. This result clearly identifies the material as susceptible and does not rely on finding part-through cracks.

8.4.4 Dead-weight testers capable of maintaining constant pressure on a hydraulic cell may be used for constant-load testing (see Figure 4).