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Item No. 21212

Standard
Test Method

Laboratory Testing of Metals for Resistance to Sulfide Stress Cracking and Stress Corrosion Cracking in H_2S 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)*[1] when operating at room temperature and *stress corrosion cracking (SCC)*[1] when operating at higher temperatures. In recognition of the variation with temperature and with different materials this phenomenon is here called *environmental cracking (EC)*.[1] For the purposes of this standard, EC includes only SSC, SCC, and hydrogen stress cracking. 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[2] standard test methods have been referenced for supplementary tests, creating a comprehensive test method standard.

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 weight-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.

This standard was prepared and revised by NACE International Task Group T-1F-9 on Metallic Materials Testing Techniques for Sulfide Corrosion Cracking, a component of Unit Committee T-1F on Metallurgy of Oilfield Equipment. The experience of Task Group T-1F-9 members with different types of stress corrosion cracking specimens for standardization is summarized and presented here. The standard was issued by NACE under the auspices of Group Committee T-1 on Corrosion Control in Petroleum Production in 1977 and revised in 1986, 1990, and 1996.

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[1] "NACE Glossary of Corrosion-Related Terms" (Houston, TX: NACE International).

[2] American Society for Testing and Materials (ASTM), 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959.

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NACE International Standard Test Method

Laboratory Testing of Metals for Resistance to Specific Forms of Environmental Cracking in H_2S Environments

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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 H_2S. 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 H_2S. 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 Safety Precautions: H_2S 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, must be noted:

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.[3])

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 environmental cracking susceptibility can be an important parameter. The fracture path in the test specimen should match what is anticipated in the actual component.

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.

[3] NACE Standard TM0284 (latest revision), "Evaluation of Pipeline and Pressure Vessel Steels for Resistance to Hydrogen-Induced Cracking" (Houston, TX: NACE).

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 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 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 (CH_3COOH), sodium acetate (CH_3COONa), 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 1193[4]). 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: Material Properties

4.1 Tensile testing in accordance with standard test methods such as ASTM A 370[5] shall be used to determine base material properties. Two or more 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 property variations that normally occur from specimen to specimen.

4.2 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.

4.3 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 specimens.

5.2 Vessels shall be capable of being purged to remove oxygen before beginning the test and of keeping air out

[4] ASTM D 1193 (latest revision), “Standard Specification for Reagent Water” (West Conshohocken, PA: ASTM).
[5] ASTM A 370 (latest revision), “Standard Test Methods and Definitions for Mechanical Testing of Steel Products” (West Conshohocken, PA: ASTM).

during the test. Using a small outlet trap on the H_2S effluent line to maintain 25 mm (1.0 in.) of water back pressure on the test vessel will prevent oxygen entry through small leaks or by diffusion up the vent line. (See Appendix B, text under "Reasons for Exclusion of Oxygen.")

5.3 Test vessels shall be sized to maintain the 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 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 vessels have not exhibited this tendency.

5.5 Test specimens shall be electrically isolated from test vessels and 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 shall consist of an acidified, H_2S-saturated aqueous environment. The solution pH after H_2S saturation but before contact with a test specimen is expected to range between 2.6 and 2.8; compare to Test Solution B, below. During the test, pH may increase but shall not exceed 4.0. If the solution-volume-to-specimen-surface-area ratios are maintained and steps are taken to exclude oxygen from the test cell as specified in this standard, the pH will not exceed this value. Test Solution A shall be used in Methods A, C, and D unless the properties of Test Solution B are required.

6.2 Test Solution B shall consist of an acidified and buffered solution having a pH between 3.4 and 3.6 after H_2S saturation but before contact with a test specimen. This solution is allowed in Methods A, C, and D. During the test the pH may increase but shall not exceed 4.0. The use of this solution shall be indicated on the material testing report. The elevated pH environment can be used when test specifications require a solution with elevated pH. For example, testing martensitic stainless steels for inclusion in NACE Standard MR0175[6] requires a test solution pH greater than or equal to 3.5.

6.3 All reagents added to the test solutions shall be measured to ±1.0% of the quantities specified for the specific test method.

6.4 The test solution shall be maintained at 24 ±3°C (75 ±5°F) unless otherwise specified in accordance with testing at elevated temperature (see Section 7). Any variations beyond this range shall be reported.

6.5 See the specific test method for the required test environment (Sections 8 through 11).

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 (14.5 psia), the following modified techniques are available.

[6] NACE Standard MR0175 (latest revision), "Sulfide Stress Cracking Resistant Metallic Materials for Oilfield Equipment" (Houston, TX: NACE).

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 here, it may not address all aspects and will need to 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 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 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 cm^3.

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 ±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.

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.

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. 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 is recommended.

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 vessel shall be sealed and leak tested. 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 vessel and risk explosion. The volume of test solution should be less than 75% of the total volume of the vessel. Moreover, a greater safety margin (smaller percentage of total volume) is recommended 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 solution for a minimum period of 1 h/L of test solution.

7.6.4 The H_2S partial pressure, pH_2S, in the test environment shall be determined by one of the following two methods:

7.6.4.1 Vessel heated before gas admitted

7.6.4.4.1 The 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.

7.6.4.4.2 Gas shall be admitted to the vessel until the test pressure, P_T, is reached.

7.6.4.4.3 The H_2S partial pressure, pH_2S, in the test environment is given approximately in Equation (1):

$$pH_2S = (P_T - P_1)\, X_{H_2S} \qquad (1)$$

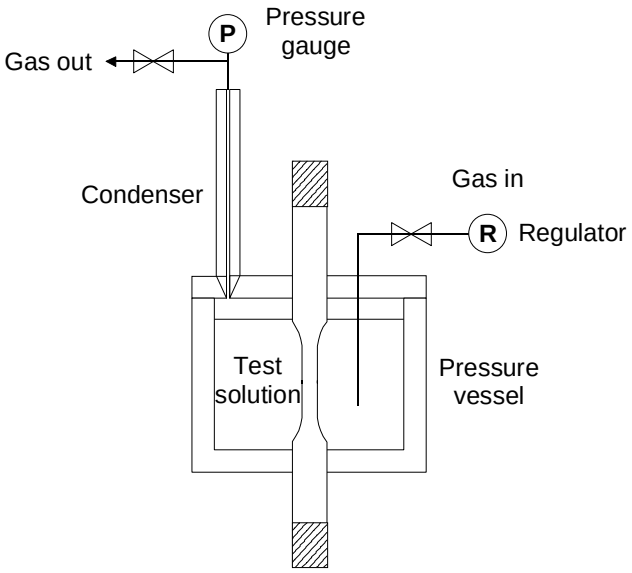


Figure 1
Schematic arrangement of test equipment for Method A - NACE Standard Tensile Test

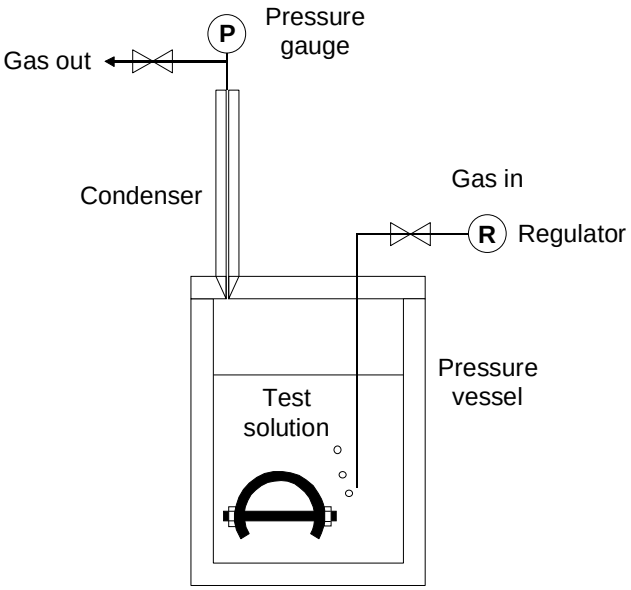


Figure 2
Schematic arrangement of test equipment for Method B - NACE Standard Bent-Beam Test Method C - NACE Standard C-Ring Test Method D - NACE Standard Double-Cantilever-Beam Test

where:
P_T = total 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 Gas admitted before vessel heated

Gas may be admitted to the vessel before heating if a proven means of calculating pH_2S can be demonstrated.

7.6.5 Gas shall be replenished as needed to maintain the required test conditions (primarily H_2S partial pressure) as outlined in Paragraph 7.2. Continuous gas bubbling at 0.5 to 1.0 cm^3/min or periodic gas replenishment once or twice weekly has been found necessary when testing CRAs at H_2S partial pressures below 2 kPa (0.3 psia) or carbon and alloy steels at H_2S partial pressures below 100 kPa (14.5 psia). Test solution loss and ingress of oxygen during 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 shall be maintained within ±3°C (±5°F) of the specified test temperature and recorded manually on a daily basis or at shorter intervals by data recorder. 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 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 specimens loaded to a particular stress level give a failure/no-failure test result. When testing multiple specimens at varying stress levels, an apparent threshold stress for EC can be obtained.[7]

[7] J.B. Greer, "Results of Interlaboratory Sulfide Stress Cracking Using the NACE T-1F-9 Proposed Test Method," MP 16, 9 (1977): p. 9.

8.1.1 This section states the procedure for testing at room temperature and atmospheric pressure. Special considerations for testing at elevated temperature and pressure are stated 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 specimen can affect the results and should be noted.

8.2.2 The gauge section of the tensile 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 test specimen with gauge section of 3.81 ±0.05 mm (0.150 ±0.002 in.) in diameter by 25.4 mm (1.00 in.) long is acceptable. After machining, test specimens should be stored in a desiccator or in uninhibited oil until ready for testing.

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 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 µin.) 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 specimen cannot be obtained from the material because of its size or shape, an appropriate subsize specimen may be used. However, subsize specimens can produce shorter failure times than those observed for standard-size specimens. The report of test data using subsize specimens should include the use and size of subsize 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 not in the gauge section.

8.2.9 Test Specimen Cleaning

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

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

8.3 Test Solution

8.3.1 Test Solution A shall consist of 5.0 wt% NaCl and 0.5 wt% glacial acetic acid dissolved in distilled or deionized water. For example, 50.0 g of NaCl and 5.0 g of glacial acetic acid can be dissolved in 945 g of distilled or deionized water. Test Solution A shall be used unless the properties of Test Solution B (below) are required.

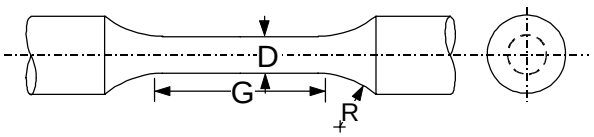
8.3.2 Test Solution B shall be used for tests requiring initial pH between 3.4 and 3.6 and shall consist of 5.0 wt% NaCl, 0.4 wt% sodium acetate (CH_3COONa), and 0.23 wt% glacial acetic acid dissolved in distilled or deionized water. The solution shall be saturated with H_2S using the same procedure as for Test Solution A.

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.

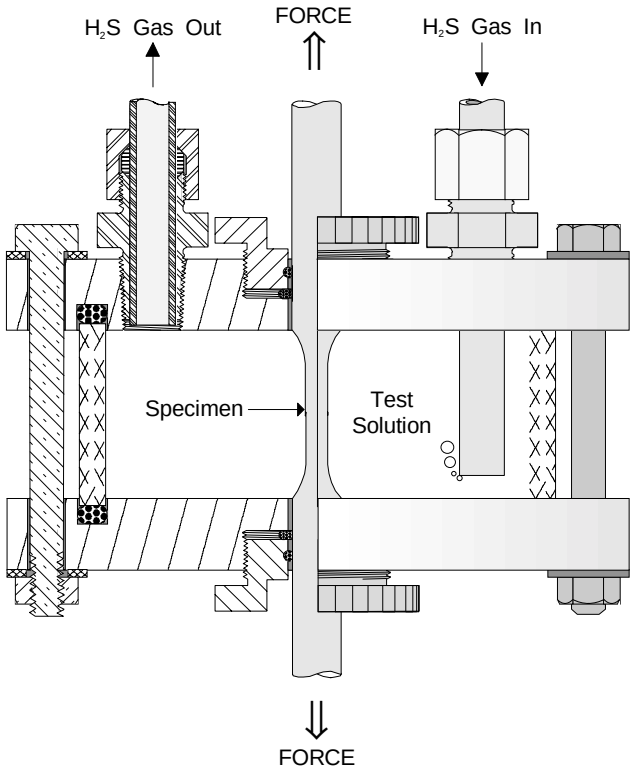
8.4.2 Tensile tests should be performed with constant-load devices or sustained-load (proof ring or spring-loaded) devices (see ASTM G 49[8]).

[8] ASTM G 49 (latest revision), "Standard Practice for Preparation and Use of Direct Tension Stress-Corrosion Test Specimens" (West Conshohocken, PA: ASTM).



Dimension	Standard specimen	Subsize 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.)	25.4 mm (1.00 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

8.4.2.1 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 can be used for constant-load testing (see Figure 4).

8.4.5 Sustained-load tests can be conducted with spring-loaded devices and proof rings when relaxation in the fixtures or test specimen will result in only a small percentage decrease in the applied load (see Figure 5).

8.4.5.1 In using proof rings, the following procedures are required:

8.4.5.1.1 Before calibration, proof rings shall be preconditioned by stressing at least 10 times to 110% of the maximum load rating of the proof ring.

8.4.5.1.2 The load on the tensile specimen shall lie within the load range of the proof ring. Accordingly, proof rings shall be selected so that the applied load will produce a ring deflection of more than 0.6% of the ring diameter but not less than 0.51 mm (0.020 in.). If it is less than 0.51 mm (0.020 in.) or less than 0.6% of the ring diameter, the calibration deflection, calibration load, and test load must be specified.

8.4.5.2 A substantial decrease in the proof ring deflection may signify:

(a) the initiation and propagation of cracks in the test specimen;

(b) yielding of the test specimen; or

(c) relaxation of stress.

Measuring the proof ring deflection during the test or at the test completion should be considered.

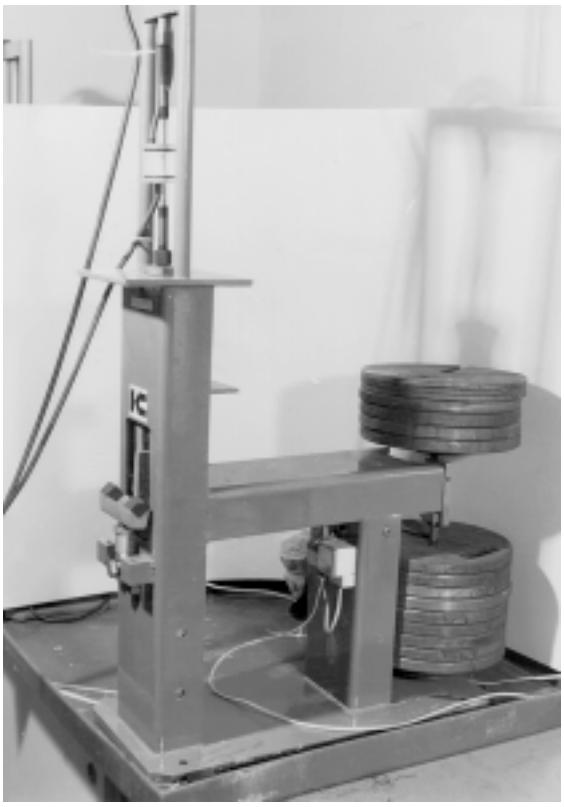
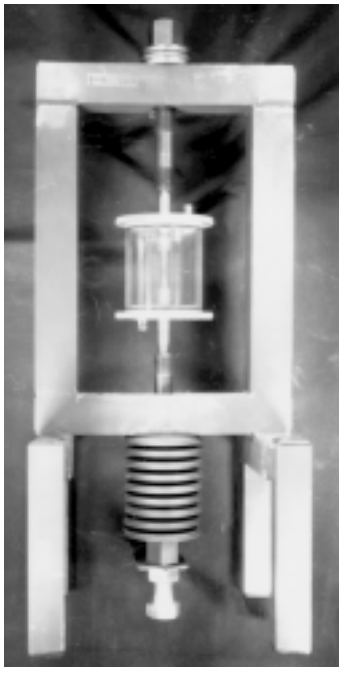


Figure 4
Constant-load (dead weight) device

(a) Proof ring



(b) Spring-loaded

Figure 5
Sustained-load devices

8.4.5.3 The deflection should be monitored when the applied stress is within 10% of the material yield strength.

8.4.6 The test specimen must be electrically isolated from any other metals in contact with the test solution.

8.4.6.1 The seals around the test specimen must be electrically isolating and airtight but should allow movement of the specimen with negligible friction.

8.4.6.2 In cases where the complete test fixture can be immersed in a test solution, the stressing fixture may be made of the same material, or, if it is made of a different material, it must be electrically isolated from the test specimen. The stressing fixture may be coated with a nonconductive impermeable coating, if desired.

8.4.7 The test vessel shall be sized to maintain a solution volume of 30 ±10 mL/cm^2 of test specimen surface area.

8.5 Stress Calculations

8.5.1 Loads for stressing tensile test specimens shall be determined from Equation (2):

$$P = S \times A \quad (2)$$

where:

P = load;
S = applied stress; and
A = actual cross-sectional area of the gauge section.

8.6 Testing Sequence

8.6.1 The minimum gauge diameter of the test specimen shall be measured, and the test specimen load shall be calculated for the desired stress level.

8.6.2 The tensile test specimen shall be cleaned and placed in the test vessel, and the test vessel shall be sealed to prevent air leaks into the vessel during the test.

8.6.3 The load may be applied before or after the test vessel is purged with inert gas.

8.6.3.1 Tensile test specimens may be stressed at convenient increments of the yield strength or load.

8.6.4 The load should be carefully applied to avoid exceeding the desired value. If the desired load is exceeded, the test shall be run at the new load or discarded.

8.6.5 The test vessel shall be immediately filled with deaerated test solution such that the solution/gas interface does not contact the gauge section of the test specimen. Deaerated solution can be prepared in a sealed vessel that is purged with inert gas at a rate of at least 100 mL/min for at least 1 h/L of solution. The solution, once in the test vessel, shall be purged with inert gas for at least 20 minutes. This inert gas purge ensures that the test solution is oxygen-free before introducing H_2S (see Appendix B). Other methods of deaeration and transfer are acceptable if the procedure results in a completely deaerated condition before H_2S introduction. Oxygen contamination is evident by a cloudy (opaque) appearance in the solution when the H_2S gas enters the test vessel. An opaque appearance to the test solution upon H_2S entry shall disqualify the test. The test specimen shall be removed and cleaned, and the test solution makeup, transfer, and deaeration procedure repeated.

8.6.6 The test solution shall then be saturated with H_2S at a rate of 100 to 200 mL/min for 20 min/L of solution. A continuous flow of H_2S through the test vessel and outlet trap shall be maintained for the duration of the test at a low flow rate (a few bubbles per minute). This maintains the H_2S concentration and a slight positive pressure to prevent air from entering the test vessel through small leaks.

8.6.7 The termination of the test shall be at test specimen failure or after 720 hours, whichever occurs first.

8.6.8 When needed, additional test specimens shall be tested to define closely the no-failure stress.

8.7 Failure Detection

Following exposure, the surfaces of the gauge section of the nonfailed test specimens shall be cleaned and inspected for evidence of cracking. Those specimens containing cracks shall be noted.

8.7.1 For all materials, failure is either:

(a) Complete separation of the test specimen; or

(b) Visual observation of cracks on the gauge section of the test specimen at 10X after completing the 720-hour test duration. Investigative techniques employing metallography, scanning microscopy, or mechanical testing may be used to determine whether cracks on the gauge section are evidence of EC. If it is verified that the cracks are not EC, then the test specimen passes the test.

8.7.2 Time-to-failure shall be recorded using electrical timers and microswitches.

8.8 Reporting of Test Results

8.8.1 Time-to-failure and no-failure data or the visual observation of surface cracks at the end of the test shall be reported for each stress level.

8.8.2 The chemical composition, heat treatment, mechanical properties, other information specified above, and data taken shall be reported.

8.8.3 Table 1 shows a suggested format for reporting the data. Data may also be presented on semilog graph paper (see Figure 6).

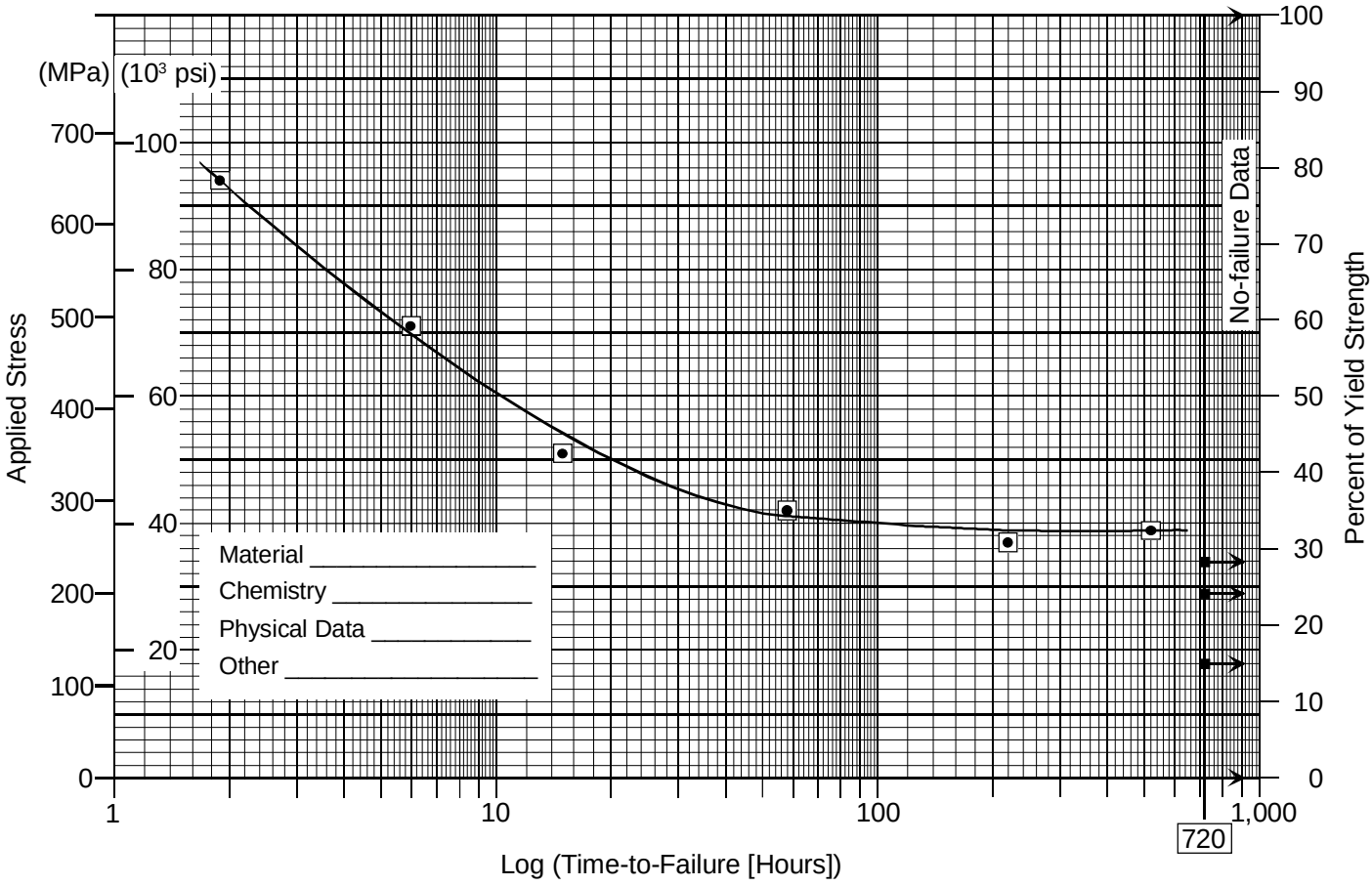


Figure 6
Applied Stress vs. Log (Time-to-Failure)

Table 1
NACE Uniform Material Testing Report Form (Part 1)
Testing per NACE TM0177-96[A]
Method A — Tensile Test

Submitting Company _____ Submittal Date _____
Submitted by _____ Telephone No. _____ Testing Lab _____
Alloy Designation _____ General Material Type _____

	Heat Number/Identification					
Chemistry						
C Mn Si P S Ni Cr Mo V Al Ti Nb N Cu Other						
Material Processing History Melt Practice (e.g., OH, BOF, EF, AOD)[B]						
Product Form						
Heat Treatment (Specify time, temperature, and cooling mode for each cycle in process.)						
Other Mechanical, Thermal, Chemical, or Coating Treatment[C]						

[A] Test method must be fully described if not in accordance with TM0177-96.
[B] Melt practice: Open-hearth (OH), Basic oxygen furnace (BOF), Electric furnace (EF), Argon-oxygen decarburization (AOD).
[C] E.g., cold work, plating, nitriding, prestrain.

Table 1
NACE Uniform Material Testing Report Form (Part 2)
Testing per NACE TM0177-96
Method A — Tensile Test

Lab Data for Material_____ Tested per NACE TM0177-96[A]

Test Specimen Geometry: ☐ Standard ☐ Nonstandard Nominal Diameter_____ Gauge Length_____
Constant Load — ☐ Dead Weight ☐ Hydraulic ☐ Other:_____
Sustained Load — ☐ Proof Ring ☐ Spring ☐ Other:_____
☐ Post-Test Proof Ring Deflection Measurement

Chemistry: ☐ Test Solution A ☐ Test Solution B ☐ Other Test Solution_____

☐ Outlet Trap to Exclude Oxygen ☐ Temperature Maintained 24° ± 3°C (75° ± 5°F) ☐ Temperature Maintained _____ ± 3°C (± 5°F)

Material Identification	Test Specimen Properties								Test Stress (% of Yield Strength)								Solution pH[E]		Applied Heat Treatment	Remarks (Including Surface Condition and H_2S Level)
	Location[B]	Orientation[C]	Yield Strength[D] ( )	Ultimate Tensile Strength ( )	Elongation (%)	Reduction in Area (%)	Hardness ( )										Start	Finish		
									Time-to-Failure (Hours) NF = No Failure at 720 Hours											

[A] Test method must be fully described if not in accordance with NACE TM0177-96.

[B] Location of test specimen from test piece may be: tubulars — outside diameter (OD), midwall (MW), or inside diameter (ID); solids — surface (S), quarter-thickness (QT), midradius (MR), center (C), or edge (E).

[C] Orientation may be longitudinal (L) or transverse (T).

[D] Open parentheses must be filled with metric or English units, as appropriate to the data. Yield strength is assumed to be 0.2% offset unless otherwise noted.

[E] Enter pH for test conducted on nonfailed specimen at highest stress if summarizing data.

Section 9: Method B — NACE Standard Bent-Beam Test

9.1 Method B, the NACE Standard Bent-Beam Test, provides for testing carbon and low-alloy steels subjected to tensile stress to evaluate resistance to cracking failure in low-pH aqueous environments containing H_2S. It evaluates EC susceptibility of these materials in the presence of a stress concentration. The compact size of the bent-beam test specimen facilitates testing small, localized areas and thin materials. Bent-beam test specimens loaded to a particular deflection give a failure/no-failure test result. When testing multiple test specimens at varying deflections, a critical stress (S_C) value can be obtained. NaCl is not added to the test solution for this test method. Laboratory test data for carbon and low-alloy steels have been found to correlate with field data.[9]

9.1.1 This section states the procedure for testing at room temperature and atmospheric pressure. Special considerations for testing at elevated temperature and pressure are stated in Section 7.

9.1.2 Method B can be summarized as follows:

9.1.2.1 This method involves deflecting each test specimen in a series by applying a bending stress. The stressed test specimens then are exposed to the test environment, and the failure (or no-failure) by cracking is determined. An S_C value that can be used to relate resistance to SSC is calculated from these data.

9.1.2.2 This method constitutes a constant-deflection test of low test specimen compliance. The computed stress is called a pseudo-stress because it does not reflect:

(a) actual stress or stress distribution in the test specimen;

(b) deviation from elasticity associated with plastic deformation; or

(c) decrease in stress in the test specimen as a crack or cracks grow.

Consequently, this method is not suitable for determination of threshold stress.

9.2 Test Specimen

9.2.1 The dimensions of the bent-beam test specimen shall be 4.57 ±0.13 mm (0.180 ±0.005 in.) wide, 1.52 ±0.13 mm (0.060 ±0.005 in.) thick, and 67.3 ±1.3 mm (2.65 ±0.05 in.) long (see Figure 7). After machining, test specimens should be stored in a desiccator or in uninhibited oil until ready for testing.

9.2.2 Generally, 12 to 16 test specimens should be taken from a given sample to determine susceptibility of the material.

9.2.2.1 The orientation and location of the test specimen with respect to the original material must be reported with the test results.

9.2.3 The test specimens should be milled to an approximate size and then surface ground to final dimensions. The last two passes on either side shall be restricted to removal of 0.013 mm (0.00050 in.) per pass (care must be taken to prevent overheating). The final surface roughness must be 0.81 μm (32 μin.) or finer.

9.2.4 As shown in Figure 7, two 0.71-mm (0.028-in.) diameter holes (No. 70 drill bit) must be drilled at the midlength of the test specimen, centered 1.59 mm (0.0625 in.) from each side edge. Holes shall be drilled before machining the final surface.

9.2.5 Test Specimen Identification

9.2.5.1 The test specimens may be stamped or vibratory stenciled in a region within 13 mm (0.50 in.) of either end on the compression side.

9.2.6 Test Specimen Cleaning

9.2.6.1 Surfaces and edges of the test specimen shall be ground by hand on 240 grit emery paper with scratches parallel to the test specimen axis.

9.2.6.2 The test specimens shall be degreased with solvent and rinsed with acetone.

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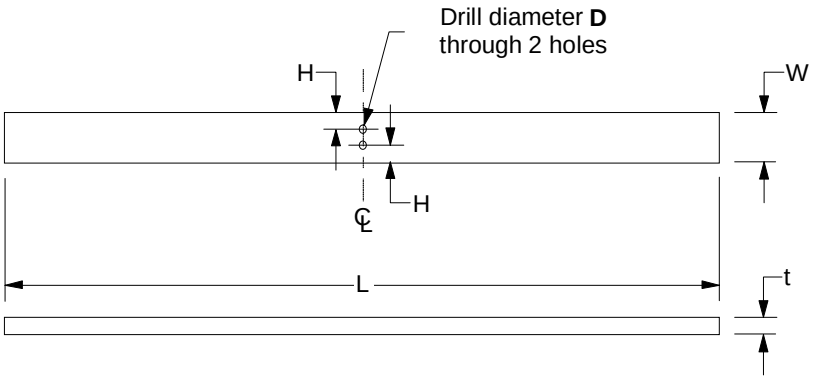
[9] J.P. Fraser, G.G. Eldridge, R.S. Treseder, "Laboratory and Field Methods for Quantitative Study of Sulfide Corrosion Cracking," H_2S Corrosion in Oil and Gas Production — A Compilation of Classic Papers, R.N. Tuttle, R.D. Kane, eds. (Houston, TX: NACE, 1981), p. 283. Original Publication: Corrosion 14, 11 (1958): p. 517t



Dimension	Size	
	(mm)	(in.)
L	67.3 ±1.3	2.65 ±0.05
t	1.52 ±0.13	0.060 ±0.005
W	4.57 ±0.13	0.180 ±0.005
H	1.59	0.0625
D	0.71	0.028 (No. 70 Drill)

Figure 7
Dimensional drawing of the standard bent-beam test specimen

9.2.6.3 The stressed section of the test specimen shall not be handled or contaminated after cleaning.

9.3 Test Solution

9.3.1 The test solution shall consist of 0.5 wt% glacial acetic acid dissolved in distilled or deionized water. For example, 5.0 g of glacial acetic acid can be dissolved in 995 g of distilled or deionized water. NaCl shall not be added to the test solution.

9.3.2 Use of Test Solutions A and B with this test method has not been standardized.

9.4 Test Equipment

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

9.4.2 Tests should be performed using constant-deflection fixtures that employ three-point bending of the test specimen. (See Figure 8.)

9.4.3 Test fixtures immersed in a test solution should resist general corrosion (UNS S31600 is commonly used). Fixture elements contacting the test specimen must be electrically isolated from it.

9.4.4 Deflection gauges shall be graduated in 0.0025-mm (0.00010-in.) divisions.

9.4.4.1 Test specimen deflection should be determined by separate gauges or by gauges incorporated in a loading fixture. In designing a deflection gauge to suit individual circumstances, the deflection at midlength of the test specimen should be measured.

9.4.5 Test Vessel

9.4.5.1 The test vessel should be sized to maintain a solution volume of 30 ±10 mL/cm^2 of test specimen surface area. A maximum volume of 10 L per test vessel is recommended.

9.4.5.2 The test vessel shall be valved at both inlet and outlet to prevent contamination of the test solution by oxygen.

9.4.5.3 A fritted glass bubbler shall be used to introduce the inert gas and H_2S below the array of test specimens. The bubbles should not impinge on the test specimens.

9.5 Deflection Calculations

9.5.1 An estimated pseudo-stress (S_C) for the material shall be used in beam deflection calculations. For carbon and low-alloy steels, S_C values are typically in the range of 69 MPa (10^4 psi) at 22 to 24 HRC. As hardness increases, S_C generally decreases.

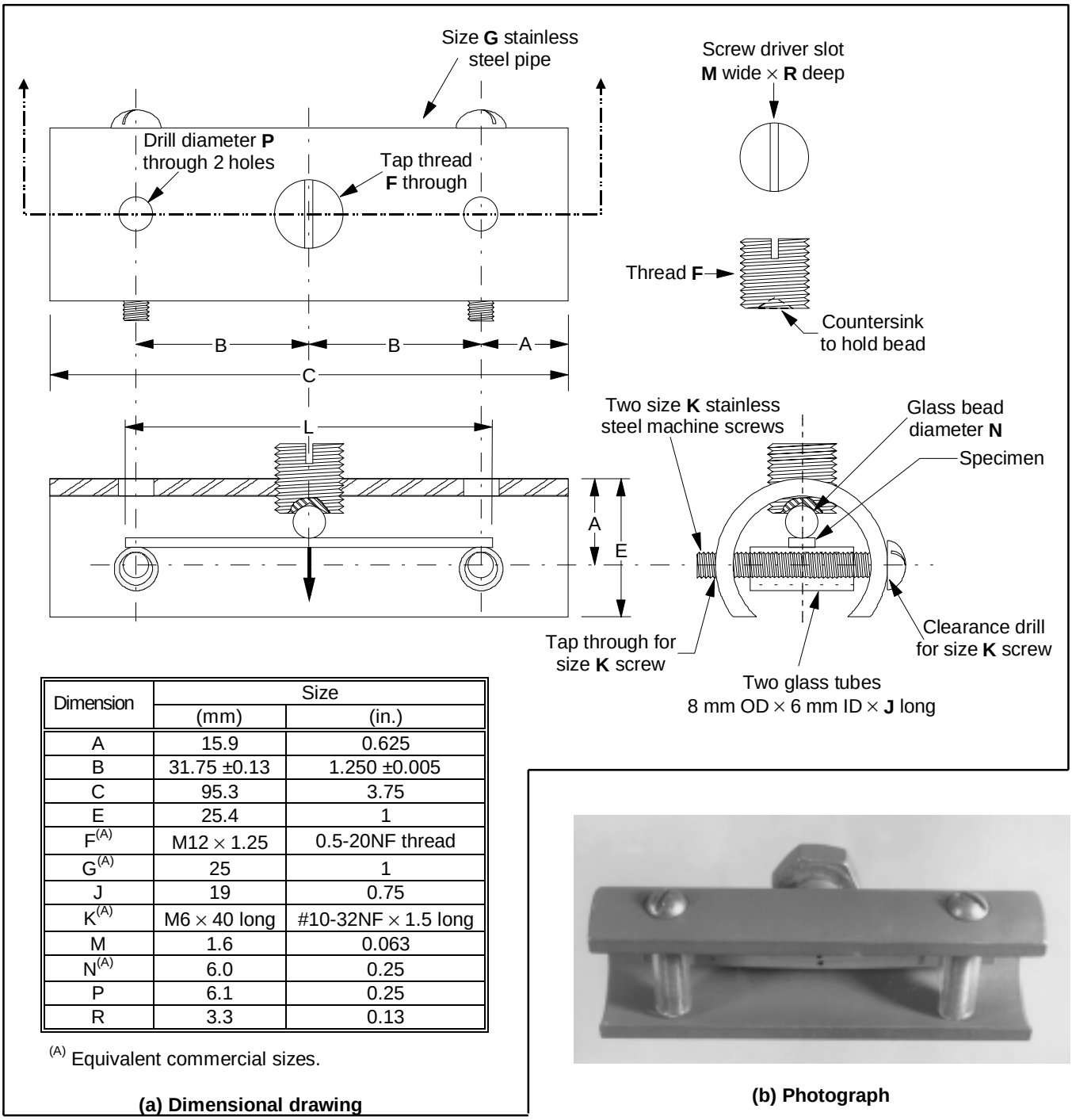
9.5.2 The selected range of estimated S_C values shall be used as pseudo-stresses to calculate the deflections of the beam specimens.

9.5.3 The specimen deflection shall be calculated for each of the pseudo-stress values using Equation (3):

$$D = \frac{SL^2}{6Et} \qquad (3)$$

where:

D = deflection;
S = pseudo-stress;
L = beam length;
E = elastic modulus; and
t = beam thickness.



Dimension	Size	
	(mm)	(in.)
A	15.9	0.625
B	31.75 ±0.13	1.250 ±0.005
C	95.3	3.75
E	25.4	1
F[(A)]	M12 × 1.25	0.5-20NF thread
G[(A)]	25	1
J	19	0.75
K[(A)]	M6 × 40 long	#10-32NF × 1.5 long
M	1.6	0.063
N[(A)]	6.0	0.25
P	6.1	0.25
R	3.3	0.13

[(A)] Equivalent commercial sizes.

(a) Dimensional drawing

(b) Photograph

Figure 8
Typical stressing fixture for bent-beam test specimen

Values for **L** and **t** are given in Figure 7. The formula assumes elastic conditions and ignores the stress concentration effect of the holes and the test specimen plasticity at high stress levels.

9.6 Testing Sequence

9.6.1 The test specimen dimensions shall be measured, and deflections shall be calculated for desired pseudo-stress levels.

9.6.2 Test specimens shall be stressed in fixtures by deflecting them to the nearest 0.0025 mm (0.00010 in.) with dial gauge and fixture.

9.6.2.1 The deflection should be carefully applied to avoid exceeding the desired value. If the desired deflection is exceeded, the test shall be run at the higher deflection or discarded.

9.6.3 The stressed test specimens shall be cleaned and placed into the test vessel.

9.6.4 The test vessel shall be filled immediately with deaerated test solution and sealed. This deaerated test solution shall be prepared in a sealed vessel that is purged with inert gas for at least 1 h/L of solution at a rate of at least 100 mL/min. The test solution, once in the test vessel, shall then be purged with inert gas for at least 20 minutes. The test solution shall then be saturated with H_2S at a rate of 100 to 200 mL/min for 20 min/L of solution.

9.6.4.1 The H_2S in the test vessel shall be replenished periodically by bubbling H_2S for a duration of 20 to 30 minutes at a rate of 100 to 200 mL/min per liter of solution three times per week for the duration of the test. The days for the replenishment should be the first, third, and fifth day of each week.

9.6.5 The test shall be terminated after 720 hours or when all specimens have failed, whichever occurs first.

9.6.6 Additional test specimens and iterative testing may be necessary to define the S_C value closely.

9.7 Failure Detection

9.7.1 Crack presence shall be determined visually with the aid of a low-power binocular microscope. If the test specimen contains only one or a few cracks, the shape of the test specimen may have changed considerably, predominantly by kinking; this feature helps to identify cracked test specimens. However, if many cracks are present, a shape change may not be apparent. Because corrosion products may obscure cracks, a careful examination is mandatory. Mechanical cleaning or metallographic sectioning of the test specimen may be necessary in these instances to detect cracks.

9.7.2 Failure is cracking of the test specimen. Consequently, following exposure, the surface of the test specimens should be cleaned and visually inspected at 10X for evidence of cracking following a 20-degree bending by hand. Test specimens found to contain cracks shall be considered failed.

9.8 Reporting of Test Results

9.8.1 Failure/no-failure data and pseudo-S_C-stress values shall be presented. Time-to-failure data are optional.

9.8.2 The critical stress (S_C) shall be calculated using Equation (4):

$$S_C(psi) = \frac{\frac{\sum S(psi)}{10^4} + 2\sum T}{n} \qquad (4)$$

where:

S = nominal outer fiber pseudo-stress in psi used to calculate the beam's deflection;
T = the test result (i.e., +1 for passing and -1 for failure); and
n = the total number of test specimens tested.

NOTE: If S_C is desired in metric units, it shall be computed first in psi units, then converted as needed.

When using this method, all pseudo-stress data that are more than ±210 MPa (±3.0 x 10^4 psi) from the value S_C x 10^4 shall be discarded.

9.8.3 The chemical compositions, heat treatment, mechanical properties, and other data taken shall be reported.

9.8.4 Table 2 shows a suggested format for reporting the data.

Section 10: Method C — NACE Standard C-Ring Test

10.1 Method C, the NACE Standard C-Ring Test, provides for evaluating the EC resistance of metals under conditions of circumferential loading (hoop stress). It is particularly suitable for making transverse tests of tubing and bar. EC susceptibility with the C-ring test specimen is usually determined by time-to-cracking during the test. C-ring test specimens, when deflected to a particular outer fiber stress level, give a failure/no-failure result. When testing multiple test specimens at varying stress levels, an apparent threshold stress for EC can be obtained.

10.1.1 This section states the procedure for testing at room temperature and atmospheric pressure.

Special considerations for testing at elevated temperature and pressure are stated in Section 7.

10.2 Test Specimen

10.2.1 An unnotched C-ring in accordance with ASTM G 38[10] shall be used. Sizes for C-rings may be varied over a wide range, but C-rings with an outside diameter (OD) of less than about 15.9 mm (0.625 in.) are not recommended because of increased difficulties in machining and decreased precision in stressing. A typical C-ring test specimen is shown in Figure 9.

10.2.2 The circumferential stress may vary across the width of the C-ring; the variation extent depends on the width-to-thickness (w/t) and diameter-to-thickness (d/t) ratios of the C-ring. The w/t ratio shall be between 2 and 10, and the d/t ratio shall be between 10 and 100.

10.2.3 The material used in the bolting fixtures shall be of the same material as that of the test specimen or be electrically isolated from the test specimen to minimize any galvanic effects, unless specific galvanic effects are desired.

10.2.4 Machining should be done in stages: the final two passes should remove a total of no more than 0.05 mm (0.002 in.) of metal, and the final cut should leave the principal surface with a finish of 0.81 µm (32 µin.) or finer. After machining, test specimens should be stored in a desiccator or in uninhibited oil until ready for testing.

10.2.4.1 A high-quality machined surface is normally used for corrosion test purposes. However, the as-fabricated surface of a tube or bar also may be evaluated by C-ring test specimens. Using any finishing process other than machining must be reported with the test data.

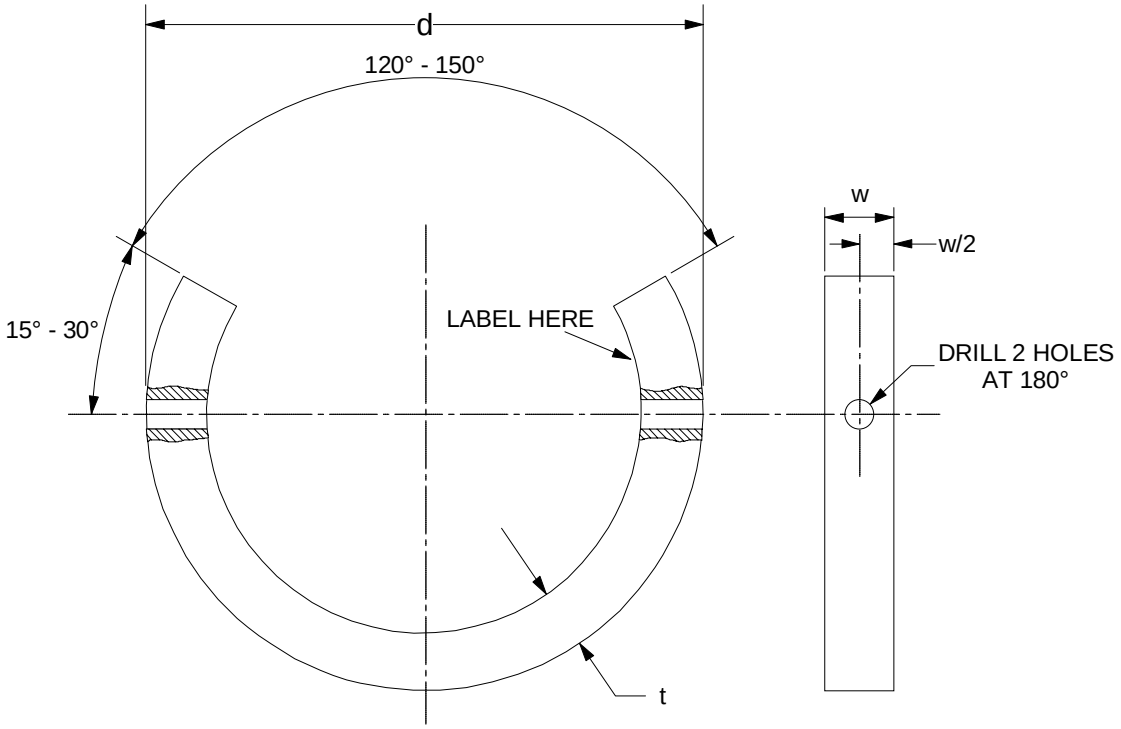


Figure 9
Dimensional drawing of the C-ring test specimen

[10]ASTM G 38 (latest revision), "Standard Practice for Making and Using C-Ring Stress-Corrosion Test Specimens" (West Conshohocken, PA: ASTM)

Table 2

NACE Uniform Material Testing Report Form (Part 1)
Testing per NACE TM0177-96[A]
Method B — Bent-Beam Test

Submitting Company _____ Submittal Date _____
Submitted by _____ Telephone No. _____ Testing Lab _____
Alloy Designation _____ General Material Type _____

Chemistry	Heat Number/Identification					
C Mn Si P S Ni Cr Mo V Al Ti Nb N Cu Other						
Material Processing History Melt Practice (e.g., OH, BOF, EF, AOD)[B]						
Product Form						
Heat Treatment (Specify time, temperature, and cooling mode for each cycle in process.)						
Other Mechanical, Thermal, Chemical, or Coating Treatment[C]						

[A] Test method must be fully described if not in accordance with TM0177-96.

[B] Melt practice: Open-hearth (OH), Basic oxygen furnace (BOF), Electric furnace (EF), Argon-oxygen decarburization (AOD).

[C] E.g., cold work, plating, nitriding, prestrain.

Table 2
NACE Uniform Material Testing Report Form (Part 2)
Testing per NACE TM0177-96
Method B — Bent-Beam Test

Lab Data for Material_____ Tested per NACE TM0177-96[A]

Test Specimen Geometry: ☐ Standard ☐ Nonstandard _____ Nominal Size _____ Length_____

Test Equipment: ☐ Statistical S_C Method Applied _____

Chemistry: ☐ Test Solution A ☐ Test Solution B ☐ Other Test Solution_____

☐ Outlet Trap to Exclude Oxygen ☐ Temperature Maintained 24° ± 3°C (75° ± 5°F) ☐ Temperature Maintained _____ ± 3°C (± 5°F)

Material Identification	Test Specimen Properties								Pseudo-Stress Values ( )								Solution pH[E]		Applied Heat Treatment	Remarks (Including Surface Condition and H_2S Level)
	Location[B]	Orientation[C]	Yield Strength[D] ( )	Ultimate Tensile Strength ( )	Elongation (%)	Reduction in Area (%)	Hardness ( )										Start	Finish		
									Time-to-Failure (Hours) NF = No Failure at 720 Hours											

[A] Test method must be fully described if not in accordance with NACE TM0177-96.

[B] Location of test specimen from test piece may be: tubulars — outside diameter (OD), midwall (MW), or inside diameter (ID); solids — surface (S), quarter-thickness (QT), midradius (MR), center (C), or edge (E).

[C] Orientation may be longitudinal (L) or transverse (T).

[D] Open parentheses must be filled with metric or English units, as appropriate to the data. Yield strength is assumed to be 0.2% offset unless otherwise noted.

[E] Enter pH for test conducted on nonfailed specimen at highest stress if summarizing data.

10.2.5 Test Specimen Identification

10.2.5.1 The test specimen end segments may be stamped or vibratory stenciled.

10.2.6 Test Specimen Cleaning

10.2.6.1 Before testing, test specimens should be degreased with solvent and rinsed with acetone.

10.2.6.2 After cleaning, the test section of the test specimen should not be handled or contaminated.

10.3 Test Solution

10.3.1 Test Solution A shall consist of 5.0 wt% NaCl and 0.5 wt% glacial acetic acid dissolved in deionized or distilled water. For example, 50.0 g of NaCl and 5.0 g of glacial acetic acid can be dissolved in 945 g of distilled or deionized water. Test Solution A shall be used unless the properties of Test Solution B (below) are required.

10.3.2 Test Solution B shall be used for tests requiring initial pH between 3.4 and 3.6 and shall consist of 5.0 wt% NaCl, 0.4 wt% sodium acetate, and 0.23 wt% glacial acetic acid dissolved in distilled or deionized water. The solution shall be saturated with H_2S using the same procedure as for Test Solution A.

10.4 Test Equipment

10.4.1 The test equipment necessary for stressing C-ring test specimens shall include calipers or equivalent equipment capable of measuring to the nearest 0.025 mm (0.0010 in.), wrenches sized to the bolting fixtures used, and a clamping device.

10.4.1.1 C-ring test specimens shall be clamped during stressing by the bolting fixtures or the tips of the C-ring. No clamping is to take place in the central test section of the C-ring.

10.4.2 The test specimen shall be so supported that nothing except the corrosive medium contacts the stressed area.

10.4.2.1 The supporting fixture shall be constructed of material compatible with the testing medium.

10.4.2.2 Galvanic effects between the test specimens, supporting fixtures, and test vessel shall be avoided. For example, an isolating bushing or washer can be used to isolate the C-ring electrically from the supporting fixtures.

10.4.3 Test Vessel

10.4.3.1 The test vessel should be sized to maintain a solution volume of 30 ±10 mL/cm^2 of test specimen surface area.

10.4.3.2 A fritted glass bubbler shall be used to introduce the inert gas and H_2S below the array of test specimens. The bubbles should not impinge on the test specimens.

10.5 Deflection Calculations

10.5.1 The deflection necessary to obtain the desired stress on the C-ring test specimen shall be calculated using Equation (5):

$$D = \frac{\pi d(d-t)S}{4tE} \quad (5)$$

where:

D = deflection of test specimen across bolt holes;
d = test specimen outer diameter;
t = test specimen thickness;
S = desired outer fiber stress; and
E = modulus of elasticity.

10.5.1.1 Deflections calculated by Equation (5) should be limited to stresses below the material elastic limit. For many CRAs the elastic limit is well below the 0.2% offset proof (yield) stress. Deflection values beyond the elastic limit can be calculated from information obtained from the stress-strain curve of the material and the strain-deflection characteristics of the specific C-ring geometry being used.

10.5.1.2 Equation (5) can be used to calculate the deflection necessary to stress the test specimen to 100% of the 0.2% offset yield strength (S_Y) by substituting $S_Y + E(0.002)$ for S in the original equation. This relationship is not valid for all alloy systems and should be checked before use on materials other than carbon and low-alloy steels.

10.5.1.3 No equation exists to calculate the deflection needed to stress test specimens to values between the material's elastic limit and its 0.2% offset proof (yield) stress.

10.5.2 The deflection can be determined directly by using electrical resistance strain gauges applied to the C-ring specimen.

10.5.2.1 Each C-ring shall be strain-gauged on the outside diameter at a point 90° opposite the axis of the C-ring bolt. The bolt shall be tightened to the appropriate strain by monitoring

the strain gauge output, then the strain gauge and glue residue shall be removed. The C-ring shall then be recleaned using the same procedures given in Paragraph 10.2.6.

10.6 Testing Sequence

10.6.1 The test specimen dimensions shall be measured, and the corresponding C-ring deflections shall be calculated.

10.6.2 C-ring test specimens shall be stressed by tightening bolting fixtures to calculated deflections measured to the nearest 0.025 mm (0.0010 in.).

10.6.2.1 Deflections shall be measured at the centerline of the bolting fixture. These measurements may be taken at the outer diameter, inner diameter, or midwall with care to maintain consistency in the points of measurement. If the desired deflection is exceeded, the test shall be run at the higher deflection or discarded.

10.6.3 The test specimens shall be cleaned and placed into the test vessel.

10.6.4 The test vessel shall be filled with deaerated test solution and sealed. This deaerated test solution shall be prepared in a sealed vessel that is purged with inert gas for at least 1 h/L of solution at a rate of at least 100 mL/min. Once in the test vessel, the test solution shall be purged with inert gas for at least 20 minutes after sealing the test vessel. The test solution shall then be saturated with H_2S at a rate of 100 to 200 mL/min for 20 min/L of solution.

10.6.5 A continuous H_2S flow through the test vessel and outlet trap shall be maintained for the duration of the test at a low flow rate (a few bubbles per minute). This maintains the H_2S concentration and a slight positive pressure to prevent air from entering the test vessel through small leaks.

10.6.6 The test duration shall be 720 hours or until all test specimens have failed, whichever occurs first.

10.7 Failure Detection

10.7.1 Highly stressed C-rings of alloys that are appreciably susceptible to EC tend to fracture through the entire thickness or to crack in a way that is conspicuous. However, with more-EC-resistant alloys, cracking frequently begins slowly and is difficult to detect. Small cracks may initiate at multiple sites and be obscured by corrosion products. It is preferable to report the first crack, if detected at 10X magnification, as the criterion of failure. An alternative method of exposing cracking in C-rings after exposure is to stress the C-ring beyond the tested stress level. Cracks resulting from EC can be differentiated from mechanically induced cracks by the corroded nature of the crack surface.

10.8 Reporting of Results

10.8.1 Failure/no-failure data shall be reported from each stress level. If time-to-failure data are recorded, they should be reported.

10.8.2 The chemical composition, heat treatment, mechanical properties, and other data taken shall be reported.

10.8.3 Table 3 shows a suggested format for reporting the data.

Section 11: Method D — NACE Standard Double-Cantilever-Beam (DCB) Test

11.1 Method D, the NACE Standard DCB Test, provides for measuring the resistance of metallic materials to propagation of EC, expressed in terms of a critical stress intensity factor, K_{ISSC} for SSC and K_{IEC} for the more general case of EC, using a crack-arrest type of fracture mechanics test. Method D does not depend on the uncertainty of pitting and/or crack initiation, because a crack is always initiated in a valid test. For SSC testing of carbon and low-alloy steels this method requires little time. Method D gives a direct numerical rating of crack propagation resistance and does not depend on evaluation of failure/no-failure results.[11] The subject of fracture mechanics testing for evaluation of EC resistance is currently under consideration by NACE Committees T-1F-9 and T-1F-9c and ASTM Committees E 8.06.02 and G 1.06.04. The user of this standard should maintain contact with these groups and their technical activities for knowledge of current state-of-the-art testing techniques.

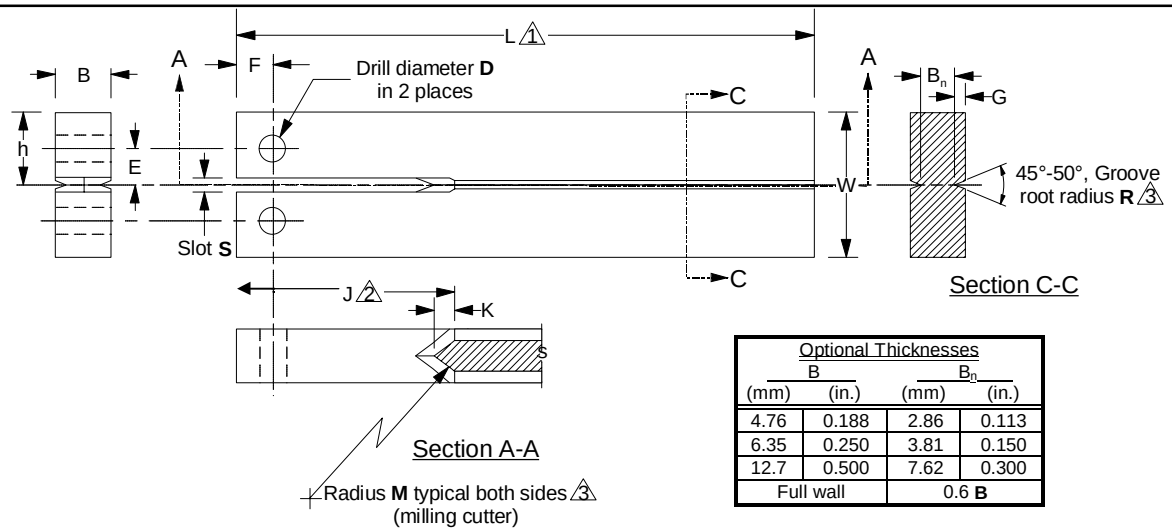
11.1.1 This section states the procedure for DCB testing at room temperature and atmospheric pressure and enables computation of K_{ISSC}. When the special considerations stated in Section 7 for testing at elevated temperature and pressure are observed, the computed stress intensity factor should be written as K_{IEC}. The equations needed to compute K_{IEC} are the same as those following shown for K_{ISSC}.

[11] R.B. Heady, "Evaluation of Sulfide Corrosion Cracking Resistance in Low Alloy Steels," Corrosion 33, 3 (1977): p. 98.

However, the following descriptions of material behavior under SSC conditions may not be accurate for the more general conditions of EC.

11.2 Test Specimen

11.2.1 The standard test specimen is designated a DCB specimen, and its design shall be as given in Figure 10(a). A double-tapered wedge shall be used to load the test specimen (see Figure 10[b]). The double-tapered wedge shall be made of the same material as the test specimen or of the same class of material as the test specimen. The wedge material may be heat-treated or cold-worked to increase its hardness and thereby help to prevent galling during wedge insertion. Wedges may be shielded with polytetrafluoroethylene (PTFE) tape to reduce corrosion in the wedge region.



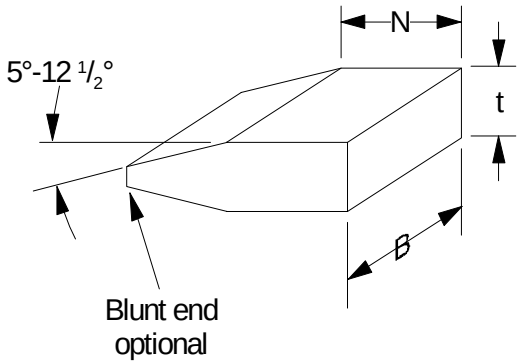
Optional Thicknesses			
B		B_n	
(mm)	(in.)	(mm)	(in.)
4.76	0.188	2.86	0.113
6.35	0.250	3.81	0.150
12.7	0.500	7.62	0.300
Full wall		0.6 **B**	

NOTES:

1 A DCB specimen of length **U** may be used if very low cracking resistance is expected.
2 A short electrodischarge-machined (EDM) slot, **X** thick, extending to a depth of **Y** from the slotted end, may be substituted for the chevron crack starter.
3 Grooves must be centered within tolerance **Z** and must be opposite within tolerance **Z**.

Dimension	Size	
	(mm)	(in.)
B	9.53 ±0.05	0.375 ±0.002
B_n	5.72 ±0.05	0.225 ±0.002
D	4.85	0.191 (No. 11 Drill)
E	6.4 +0.2/ -0.0	0.25 +0.01/ -0.00
F	6.35 ±0.10	0.250 ±0.004
G	1.91 ±0.05	0.075 ±0.002
h	12.70 ±0.05	0.500 ±0.002
J	38.10 ±1.59	1.500 ±0.063
K	3.17 ±0.77	0.125 ±0.031
L	101.60 ±1.59	4.000 ±0.063
M	51 ±13	2.0 ±0.5
N	6.35 ±0.10	0.250 ±0.004
R	0.25 ±0.05	0.010 ±0.002
S	2.38 ±0.05	0.094 ±0.002
U	130	5
W	25.40 ±0.05	1.000 ±0.002
X	0.3	0.01
Y	41.3	1.63
Z	±0.05	±0.002

(a) Design



NOTE: **t** is sufficient to give the specified arm displacement.

(b) Double-tapered wedge

Figure 10
DCB Specimen

Table 3

NACE Uniform Material Testing Report Form (Part 1)
Testing per NACE TM0177-96[A]
Method C — C-Ring Test

Submitting Company _____ Submittal Date _____
Submitted by _____ Telephone No. _____ Testing Lab _____
Alloy Designation _____ General Material Type _____

Chemistry	Heat Number/Identification					
C Mn Si P S Ni Cr Mo V Al Ti Nb N Cu Other						
Material Processing History Melt Practice (e.g., OH, BOF, EF, AOD)[B]						
Product Form						
Heat Treatment (Specify time, temperature, and cooling mode for each cycle in process.)						
Other Mechanical, Thermal, Chemical, or Coating Treatment[C]						

[A] Test method must be fully described if not in accordance with TM0177-96.
[B] Melt practice: Open-hearth (OH), Basic oxygen furnace (BOF), Electric furnace (EF), Argon-oxygen decarburization (AOD).
[C] E.g., cold work, plating, nitriding, prestrain.

Table 3
NACE Uniform Material Testing Report Form (Part 2)
Testing per NACE TM0177-96
Method C — C-Ring Test

Lab Data for Material_____ Tested per NACE TM0177-96[A]

Test Specimen Geometry: Outside Diameter _____ Wall/Thickness _____ Width_____

Test Equipment: ☐ Bolting Material Same as Specimen
☐ Correction for Yielding Applied

Chemistry: ☐ Test Solution A ☐ Test Solution B ☐ Other Test Solution_____

☐ Outlet Trap to Exclude Oxygen ☐ Temperature Maintained 24° ± 3°C (75° ± 5°F) ☐ Temperature Maintained _____ ± 3°C (± 5°F)

Material Identification	Test Specimen Properties								Applied Stress (% of Yield Strength)								Solution pH[E]		Applied Heat Treatment	Remarks (Including Surface Condition and H_2S Level)
	Location[B]	Orientation[C]	Yield Strength[D] ( )	Ultimate Tensile Strength ( )	Elongation (%)	Reduction in Area (%)	Hardness ( )										Start	Finish		
									Time-to-Failure (Hours) NF = No Failure at 720 Hours											

[A] Test method must be fully described if not in accordance with NACE TM0177-96.

[B] Location of test specimen from test piece may be outside diameter (OD), midradius (MR), inside diameter (ID), or full wall (FW).

[C] Orientation may be longitudinal (L) but is usually transverse (T).

[D] Open parentheses must be filled with metric or English units, as appropriate to the data. Yield strength is assumed to be 0.2% offset unless otherwise noted.

[E] Enter pH for test conducted on nonfailed specimen at highest stress if summarizing data.

11.2.2 The standard test specimen thickness shall be nominally 9.53 mm (0.375 in.); complete dimensions are shown in Figure 10(a). When the material being tested is too thin to meet this requirement, other thicknesses as noted in the figure may be considered. Subsized test specimens of some carbon and low-alloy steels may give lower K_{Issc} values than standard-sized specimens; differences as high as 37% have been observed. Further work is necessary to quantify this effect.

11.2.3 Full-thickness test specimens may be prepared from tubular products if the ratio of the tubular outside diameter to the wall thickness is greater than 10. The side grooves should be 20% of the wall thickness, thus maintaining a web thickness ($\mathbf{B}_n$) equal to 60% of the wall thickness.

11.2.4 The side grooves must be machined carefully to avoid overheating and cold working. The final two machining passes should remove a total of 0.05 mm (0.002 in.) of metal. Grinding is also acceptable if the process does not harden the material.

11.2.5 In testing materials of low K_{Issc} (below 22 to 27 MPa$\sqrt{m}$ [20 to 25 ksi$\sqrt{in.}$]) or materials in which crack initiation is difficult, e.g., lower-yield-strength materials, introducing the electrodischarge-machined (EDM) slot noted in Figure 10(a) or a fatigue precrack is very helpful in avoiding side-cracking and in initiating SSC, respectively.

11.2.5.1 Fatigue precracking can be conducted under load control at 30 Hz, with the load varying from 310 to 3,100 N (70 to 700 lb) for the standard-sized test specimen. To avoid residual compressive stress, the peak K_I during precracking should not exceed 70% of the expected initial K_I imparted by the wedge.

11.2.6 Test Specimen Identification

11.2.6.1 Each sidearm of the DCB specimen should be identified by stamping or vibratory stenciling, either near the two holes or on the end that is not wedge-loaded.

11.2.7 Dimensional Check

11.2.7.1 Dimensions **B**, $\mathbf{B_n}$, 2**h**, and the distance of the hole centers from the near end of the test specimen shall be measured. (A blade micrometer should be used for measuring $\mathbf{B_n}$.) Any values that lie outside the limits shown in Figure 10(a) shall be recorded for later use in computing K_{Issc} (see Paragraph 11.6.3).

11.2.8 Test Specimen Cleaning

11.2.8.1 Test specimens should be degreased with solvent and rinsed in acetone.

11.3 Test Solution

11.3.1 Test Solution A shall consist of 5.0 wt% NaCl and 0.5 wt% glacial acetic acid dissolved in deionized or distilled water. For example, 50.0 g of NaCl and 5.0 g of glacial acetic can be dissolved in 945 g of distilled or deionized water. Although the initial pH is not specified, it is expected to range from 2.6 to 2.8. Test Solution A shall be used unless the properties of Test Solution B (below) are required.

11.3.2 Test Solution B shall consist of 5.0 wt% NaCl, 0.4 wt% sodium acetate, and 0.23 wt% glacial acetic acid dissolved in deionized or distilled water. The initial pH of this solution shall be between 3.4 and 3.6.

NOTE: For materials with high cracking resistance, use of fatigue precracking and/or higher initial arm displacements (δ) is recommended when using the higher-pH environment.

11.3.3 Subsequently, the test solution (Test Solution A or B) shall be saturated with H_2S as described in Paragraph 11.5.7.

11.4 Test Equipment

11.4.1 A test vessel sized to accommodate one liter of test solution per test specimen (test solution volume of 10 to 15 mL/cm^2 of test specimen surface area) should be employed. The maximum volume of the vessel should be 10 L for ease of solution saturation.

11.4.2 A slotted base plate or other test specimen holder (electrically isolating) is recommended for ensuring uniform spacing and orientation of the test specimens.

11.4.3 A small wedge-loading fixture may be attached to the jaws of a bench vise to facilitate full wedge insertion, flush with the end of the test specimen.

11.4.4 A fritted glass bubbler shall be used to introduce the inert gas and H_2S below the array of specimens. The bubbles should not impinge on the test specimens.

11.5 Testing Sequence

11.5.1 Clean the test specimens as indicated in Paragraph 11.2.8.1.

11.5.2 Prepare DCB specimens with precracks (if desired) using the method given in Paragraph 11.2.5.1.

11.5.3 Measure the slot thickness. Feeler gauges are recommended for this measurement. Because initial stress intensity affects the final K_{ISSC} of carbon and low-alloy steels,[12] wedge thicknesses for the SSC-resistant grades of such steels that achieve the arm displacements in Table 4 should be selected. The arm displacement for each grade shall not exceed the upper limit listed in Table 4 for that grade.

The acceptable arm displacements given in Table 4 also may provide guidance in the selection of wedges for testing other grades of carbon and low-alloy steels. Table 5 provides guidance in choosing wedge thicknesses for other low-alloy steels and alloys. When testing steels with yield strengths below 550 MPa (80 ksi), the arm displacement (δ) may be computed using Equation (6), in which YS is yield strength:

$$\delta(\text{mm}) = \left[2.02 - \frac{\text{YS(MPa)}}{530} \pm 0.13 \text{ mm}\right] \quad (6a)$$

$$\delta(\text{in.}) = \left[0.080 - \frac{\text{YS(ksi)}}{1{,}953} \pm 0.005 \text{ in.}\right] \quad (6b)$$

11.5.3.1 Measure the initial spacing of the loading holes in the test specimen for use in determining actual arm displacement.

11.5.3.2 Select a wedge thickness that would give an expected displacement in the middle of the desired range, because exact arm displacements are difficult to achieve.

11.5.3.3 Press the wedge into the slot, flush with the end of the test specimen.

11.5.3.4 Remeasure the spacing of the holes, and then compute the actual arm displacement (δ). For the SSC-resistant carbon and low-alloy steels in Table 5, if the actual arm displacement lies below the intended range, a new wedge may be inserted to achieve an acceptable displacement. If the actual arm displacement lies above the intended range, the wedge shall be removed, and the EDM slot or fatigue precrack shall be extended through the zone of plastically deformed material before reloading the test specimen.

11.5.4 Degrease the test specimens with solvent and rinse in acetone.
NOTE: Fatigue-precracked test specimens shall be cleaned by wiping, not by using immersion.

11.5.5 Place the test specimens in the test vessel.

11.5.6 Add deaerated test solution to the test vessel. This deaerated test solution can be prepared in a sealed vessel that is purged with inert gas at a rate of at least 100 mL/min for at least 1 h/L of solution. Once inside the test vessel, the test solution shall be purged with inert gas for at least 20 minutes after sealing the test vessel. The purpose of this inert gas purge is to ensure that the test solution is oxygen-free before introducing H_2S (see Appendix B). Other methods of deaeration and transfer are acceptable if the procedure results in a completely deaerated condition prior to H_2S introduction. Oxygen contamination is evident by a cloudy (opaque) appearance in the test solution when the H_2S gas enters the test vessel. An opaque appearance to the test solution upon H_2S entry shall disqualify the test. The test specimen shall be removed and cleaned, and the test solution makeup, transfer, and deaeration procedure repeated.

11.5.7 Saturate the test solution with H_2S at a rate of at least 100 mL/min for at least 20 min/L of test solution. A continuous H_2S flow through the test vessel and outlet trap shall be maintained for the duration of the test at a low flow rate. This maintains the H_2S concentration and a slight positive pressure to prevent air from entering the test vessel through small leaks.

11.5.8 The test duration for carbon and low-alloy steels shall be 14 days. For stainless steels, Ni-, Ni/Co-, Ti-, or Zr-base alloys, a longer test may be required to ensure that the crack has stopped growing. For these materials the test duration should be at least 30 days. The duration of the test should be mutually agreed upon among all interested parties and should be reported with the results.

11.5.9 Remove corrosion products from the test specimen by vapor honing, by bead- or sand-blasting lightly, or by any other means that does not remove a significant amount of metal.

11.5.10 Obtain the load-vs-displacement curve and remove the wedge. The equilibrium wedge-load (P) is located at the abrupt change in slope of this "lift-off" curve.

11.5.11 After wedge removal, open the DCB specimen mechanically to expose the crack surfaces.

[12] D.L. Sponseller, "Interlaboratory Testing of Seven Alloys for SSC Resistance by the Double Cantilever Beam Method," Corrosion 48, 2 (1992): p. 159.

Table 4
Arm Displacements For API[13] and Other Grade Oilfield Tubular Steels

Grade[A]	Yield Strength Range		Acceptable Arm Displacement (δ)	
	MPa	(ksi)	mm	(0.001 in.)
L-80	552-655	(80-95)	0.71-0.97	(28-38)
C-90	621-724	(90-105)	0.64-0.89	(25-35)
C-95, T-95	655-758	(95-110)	0.58-0.84	(23-33)
Grade 100[B]	689-793	(100-115)	0.51-0.76	(20-30)
Grade 105[B]	724-827	(105-120)	0.46-0.71	(18-28)
Grade 110[B]	758-862	(110-125)	0.38-0.64	(15-25)
P-110	758-965	(110-140)	0.25-0.64	(10-25)
Q-125	862-1,030	(125-150)	0.25-0.51	(10-20)

[A] API grades unless noted otherwise.
[B] Non-API grades.

Table 5
Suggested Arm Displacements for Selected Alloys and Strength Levels

Material Yield Strength		Arm Displacement (δ), mm (0.001 in.)[A]									
MPa	(ksi)	Low-Alloy Steels[B]		CA-6NM		Duplex Stainless Steels		C-276		Ti-3-8-6-4-4	
552	(80)	0.71-1.07	(28-42)	0.64-0.89	(25-35)	—		—		—	
621	(90)	0.58-0.89	(23-35)	0.46-0.71	(18-28)	—		—		—	
689	(100)	0.48-0.79	(19-31)	0.20-0.46	(8-18)	—		—		—	
827	(120)	0.33-0.58	(13-23)	—		0.89-1.02	(35-40)	—		—	
965	(140)	0.20-0.46	(8-18)	—		0.64-0.89	(25-35)	—		1.02-1.52	(40-60)
1,100	(160)	0.18-0.38	(7-15)	—		—		1.27-1.78	(50-70)	—	
1,240	(180)	0.15-0.30	(6-12)	—		—		1.02-1.27	(40-50)	—	

[A] These values apply at the indicated yield strength, not over a range of yield strengths. Therefore, the user should interpolate or extrapolate to the actual yield strength of the alloy being used.
[B] For oilfield tubular steels, use Table 4.

This procedure may require the use of a tensile machine. For ferritic steels this process can be made easier by chilling the test specimen in liquid nitrogen and splitting the arms apart with a hammer and chisel.

11.6 Determination of K_{ISSC}

11.6.1 The fracture surface shall be examined for the following characteristics:

(a) The fracture surface should show at least 2.5 mm (0.10 in.) crack growth beyond the base of the chevron, EDM slot, or fatigue precrack.

(b) If the fracture is nonplanar at the crack front, the convex surface should not project beyond the "V" portion of the side groove.

(c) The crack front should not be pinned; causes of pinning include internal cracks and fissures.

(d) The crack front should be free of disturbance by a secondary crack that converges on the crack plane from the thicker portion of the test specimen.

[13] American Petroleum Institute (API), 1220 L St. NW, Washington, DC 20005.

(e) The crack should not branch into one or both of the sidearms (side crack).

(f) The crack front should be at least 25 mm (1.0 in.) from the unslotted end.

(g) The crack front of full-thickness (curved) test specimens must not lead on the edge of the fracture surface closer to the tubular internal surface.

11.6.2 If the fracture surface is satisfactory with respect to all of the items in Paragraph 11.6.1, the test shall be considered valid. The distance from the slotted end of the test specimen to the mean position of the crack front shall be measured using a dial caliper. From that distance 6.35 mm (0.250 in.) shall be subtracted to obtain the crack length (**a**). The location of the SSC/brittle (overload) fracture boundary may be checked, if in doubt, by a staining technique before sidearm separation or scanning electron microscopy after separation.

11.6.3 The stress intensity factor for SSC of flat test specimens shall be calculated using Equation (7):

$$K_{Issc}\text{ (flat DCB specimen)} = \frac{Pa(2\sqrt{3}+2.38h/a)(B/B_n)^{1/\sqrt{3}}}{Bh^{3/2}} \qquad (7)$$

where:

K_{Issc} = threshold stress intensity factor for SSC;
P = equilibrium (final) wedge load, measured in the loading plane;
a = crack length, as described in Paragraph 11.6.2;
h = height of each arm;
B = test specimen thickness; and
B_n = web thickness.

(For any dimension that was found in Paragraph 11.2.7.1 to lie outside the specified tolerance, this actual dimension, rather than the nominal, shall be used to compute K_{Issc}.)

11.6.3.1 Calculation check:

Data: B = 9.53 mm (0.375 in.);
B_n = 5.72 mm (0.225 in.);
h = 12.7 mm (0.500 in.);
P = 1870 N (421 lbf);
a = 46.48 mm (1.830 in.); and
K_{Issc} = 35.3 MPa√m (32.1 ksi√in.).

11.6.4 The stress intensity factor for SSC of full-wall thickness (curved) specimens shall be computed using Equation (8)[14]:

$$K_{Issc}\text{ (curved DCB specimen)} = \left(\frac{3I}{Bh^3}\right)^{1/2} K_{Issc}\text{ (flat DCB specimen)} \qquad (8)$$

where:

K_{Issc} (flat DCB specimen) is calculated using Equation (7); and

Quantity **I** is computed from Equation (9) as follows:

$$I = -\frac{h}{4}\left(\frac{D^2}{4}-h^2\right)^{3/2} + \frac{D^2 h}{32}\left(\frac{D^2}{4}-h^2\right)^{1/2} + \frac{D^4}{128}\sin^{-1}\left(\frac{2h}{D}\right) + \frac{h}{4}\left[\left(\frac{D}{2}-B\right)^2-h^2\right]^{3/2} - \frac{h}{8}\left(\frac{D}{2}-B\right)^2\left[\left(\frac{D}{2}-B\right)^2-h^2\right]^{1/2} - \frac{1}{8}\left(\frac{D}{2}-B\right)^4\sin^{-1}\left(\frac{2h}{D-2B}\right) \qquad (9)$$

where:

B and **h** have values per Paragraph 11.6.3; and

D = tubular outside diameter.

Mathematical analysis and experimental results have shown that the correction factor for curved specimens results in only a few percentage increase, at most, in K_{Issc} value.

11.6.4.1 Calculation Check:

Data:

D = 193.7 mm (7.625 in.); and

Other data as in Paragraph 11.6.3.1.

[14] S. W. Ciaraldi, "Application of a Double Cantilever Beam Specimen to Stress-Corrosion Evaluation of High-Alloy Production Tubulars," CORROSION/83, paper no. 162 (Houston, TX: NACE, 1983).

Result:

K_{Issc} = 35.4 MPa√m (32.2 ksi√in.)

11.7 Reporting of Test Results:

11.7.1 For each set of test specimens, all individual values of K_{Issc} for valid tests shall be reported. The arm displacement for each test specimen shall be given.

11.7.2 The chemical composition, heat treatment, mechanical properties, and other data taken shall be reported.

11.7.3 Table 6 shows a suggested format for reporting the data.

Appendix A
Safety Considerations When Handling H_2S

H_2S is perhaps responsible for more industrial poisoning accidents than any other single chemical. A number of these accidents have been fatal. H_2S should be handled with caution, and any experiments using it should be planned carefully. The maximum allowable Occupational Safety and Health Administration (OSHA)[15] concentration in the air for an 8-hour workday is 20 parts per million (ppm), well above the level detectable by smell. However, the olfactory nerves can become deadened to the odor after exposure of 2 to 15 minutes, depending on concentration, so that odor is not a reliable alarm system.

Briefly, the following are some of the human physiological reactions to various concentrations of H_2S. Exposure to concentrations in the range of 150 to 200 ppm for prolonged periods may cause edema of the lungs. Nausea, stomach distress, belching, coughing, headache, dizziness, and blistering are signs and symptoms of poisoning in this range of concentration. Pulmonary complications, such as pneumonia, are strong possibilities from such exposure. At 500 ppm, unconsciousness usually occurs within 30 minutes and results in acute toxic reactions. In the 700 to 1,000 ppm range, unconsciousness may occur in less than 15 minutes and death within 30 minutes. At concentrations above 1,000 ppm, a single lungful may result in instantaneous unconsciousness, with death quickly following due to complete respiratory failure and cardiac arrest.

Additional information on the toxicity of H_2S can be obtained by consulting the Material Safety Data Sheet provided by the manufacturer or distributor and from consulting sources such as *Dangerous Properties of Industrial Materials* by N. Irving Sax,[16] *Documentation of the Threshold Limit Values,*[17] and the *NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards.*[18]

Fire and Explosion Hazards

H_2S is a flammable gas, yielding poisonous sulfur dioxide as a combustion product. In addition, its explosive limits range from 4.0 to 46% in air. Appropriate precautions should be taken to prevent these hazards from developing.

Experimental Suggestions

All tests should be performed in a hood with adequate ventilation to exhaust all H_2S. The H_2S flow rates should be kept low to minimize the quantity exhausted. A 10% caustic absorbent solution for effluent gas can be used to further minimize the quantity of H_2S gas exhausted. This solution will need periodic replenishment. Provision should be made to prevent backflow of the caustic solution into the test vessel if the H_2S flow is interrupted. Suitable safety equipment should be used when working with H_2S.

Particular attention should be given to the output pressure on the pressure regulators because the downstream pressure frequently rises as corrosion product, debris, and other obstructions accumulate and interfere with regulation at low flow rates. Gas cylinders should be securely fastened to prevent tipping and breakage of the cylinder head. Because H_2S is in liquid form in the cylinders, the consumption of the contents should be measured by weighing the cylinder. The pressure gauge on the cylinder should also be checked frequently, because relatively little time will elapse after the last liquid evaporates until the pressure drops from 1.71 MPa (250 psi) to atmospheric pressure. The cylinder should be replaced by the time it reaches 0.52 to 0.69 MPa (75 to 100 psi) because the regulator control may become erratic. Flow should not be allowed to stop

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[15] OSHA Rules and Regulations (Federal Register, Vol. 37, No. 202, Part II, October 18, 1972).
[16] N. Irving Sax, Dangerous Properties of Industrial Materials (New York, NY: Reinhold Book Corp., 1984).
[17] Documentation of the Threshold Limit Values (Cincinnati, OH: American Conference of Governmental Industrial Hygienists, Inc., 1969).
[18] NIOSH/OSHA, Occupational Health Guidelines for Chemical Hazards, Publication NU 81-123 (Washington, DC: Superintendent of Documents, U.S. Government Printing Office).

Table 6

NACE Uniform Material Testing Report Form (Part 1)
Testing per NACE TM0177-96[A]
Method D — Double-Cantilever-Beam Test

Submitting Company _____ Submittal Date _____
Submitted by _____ Telephone No. _____ Testing Lab _____
Alloy Designation _____ General Material Type _____

Chemistry	Heat Number/Identification					
C Mn Si P S Ni Cr Mo V Al Ti Nb N Cu Other						
Material Processing History Melt Practice (e.g., OH, BOF, EF, AOD)[B]						
Product Form						
Heat Treatment (Specify time, temperature, and cooling mode for each cycle in process.)						
Other Mechanical, Thermal, Chemical, or Coating Treatment[C]						

[A] Test method must be fully described if not in accordance with TM0177-96.
[B] Melt practice: Open-hearth (OH), Basic oxygen furnace (BOF), Electric furnace (EF), Argon-oxygen decarburization (AOD).
[C] E.g., cold work, plating, nitriding, prestrain.

Table 6
NACE Uniform Material Testing Report Form (Part 2)
Testing per NACE TM0177-96
Method D — Double-Cantilever-Beam Test

Lab Data for Material_____ Tested per NACE TM0177-96[A]

DCB Specimen Geometry: ☐ Standard ☐ Nonstandard: Thickness _____ Height (2h) _____ Length_____
☐ Galvanic Couple _____ ☐ Fatigue Precrack ☐ EDM Crack Starter

Chemistry: ☐ Test Solution A ☐ Test Solution B ☐ Other Test Solution _____

☐ Outlet Trap to Exclude Oxygen ☐ Temperature Maintained 24° ± 3°C (75° ± 5°F) ☐ Temperature Maintained _____ ± 3°C (± 5°F)

Material Identification	Location[B]	Orientation[C]	DCB Specimen Properties[D]					Data for Valid Tests										Solution pH		Remarks (Including Surface Condition and H_2S Level)
								Arm Displacement (δ) ( )				$K_{I_{SSC}}$ or $K_{I_{EC}}$ ( )								
			Yield Strength ( )	Ultimate Tensile Strength ( )	Elongation (%)	Reduction in Area (%)	Hardness ( )	1	2	3	4	1	2	3	4	Mean	Std. Dev.	Start	Finish	

[A] Test method must be fully described if not in accordance with NACE TM0177-96.

[B] Tubulars: outside diameter (OD), midwall (MW), or inside diameter (ID); solid pieces: surface (S), quarter-thickness (QT), midradius (R), or center (C).

[C] Orientation may be longitudinal (L) or transverse (T).

[D] Open parentheses must be filled with metric or English units, as appropriate to the data. Yield strength is assumed to be 0.2% offset unless otherwise noted.

without closing a valve or disconnecting the tubing at the test vessel because the solution will continue to absorb H_2S and move upstream into the flowline, regulator, and even the cylinder. A check valve in the line should prevent the problem if the valve works properly. However, if such an accident occurs, the remaining H_2S should be vented as rapidly and safely as possible, and the manufacturer should be notified so that the cylinder can receive special attention.

Appendix B
Explanatory Notes on EC Test Method

Reasons for Reagent Purity

Water impurities of major concern are alkaline- or acid-buffering constituents that may alter the pH of the test solution and organic and inorganic compounds that could change the nature of the corrosion reaction. Oxidizing agents could also convert part of the H_2S to soluble products, such as polysulfides and polythionic acids, which may also affect the corrosion process.

Alkaline materials (such as magnesium carbonate and sodium silica aluminate) are often added to (or not removed from) commercial grades of sodium chloride to assure free-flowing characteristics and can greatly affect the pH.

Trace oxygen impurities in the purge gas are much more critical than water impurities if nitrogen (or other inert gas) is continuously mixed with H_2S to obtain a lower partial pressure of H_2S in the gas and hence a lower H_2S concentration in solution. Oxidation products could accumulate, resulting in changes in corrosion rate and/or hydrogen entry rate (see the paragraph below on Reasons for Exclusion of Oxygen).

Preparation of Specimen

All machining operations should be performed carefully and slowly so that overheating, excessive gouging, and cold work do not alter critical physical properties of the material. Uniform surface condition is critical to consistent SSC test results.

Reasons for Exclusion of Oxygen

Obtaining and maintaining an environment with minimum dissolved oxygen contamination is considered very important because of significant effects noted in field and laboratory studies:

(1) Oxygen contamination in brines containing H_2S can result in drastic increases in corrosion rates by as much as two orders of magnitude. Generally, the oxygen can also reduce hydrogen evolution and entry into the metal. Systematic studies of the parameters affecting these phenomena (as they apply to EC) have not been reported in the literature.

(2) Small amounts of oxygen or ammonium polysulfide are sometimes added to aqueous refinery streams in conjunction with careful pH control near 8 to minimize both corrosion and hydrogen blistering. The effectiveness is attributed to an alteration of the corrosion product.

In the absence of sufficient data to define and clarify the effects of these phenomena on EC, all reasonable precautions to exclude oxygen should be taken. The precautions cited in this standard will minimize the effects of oxygen with little increase in cost, difficulty, or complexity.

Cautionary Notes

Cleaning solvents such as 1,1,1-trichloroethane, acetone, and other hydrocarbon liquids can be hazardous if the vapors are inhaled or absorbed through the skin. Many chlorinated hydrocarbon compounds are suspected of being carcinogenic and should be used only with the proper safeguards.

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