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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) when operating at room temperature and stress corrosion cracking (SCC) when operating at higher temperatures. In recognition of the variation with temperature and with different materials this phenomenon is herein called environmental cracking (EC). For the purposes of this standard, EC includes only SSC, SCC, and hydrogen stress cracking (HSC).

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

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

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

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

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

This standard was originally published in 1977 by NACE International Task Group T-1F-9, a component of Unit Committee T-1F, "Metallurgy of Oilfield Equipment." The standard was revised in 1986, 1990, and 1996 by Task Group T-1F-9. It was revised in 2005 and 2016 by Task Group (TG) 085, "Sulfide Corrosion Cracking: Metallic Materials Testing Techniques." TG 085 is administered by Specific Technology Group (STG) 32, "Oil and Gas Production—Metallurgy," and is sponsored by STG 62, "Corrosion Monitoring and

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

Measurement—Science and Engineering Applications." The standard is issued by NACE under the auspices of STG 32.

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Standard Test Method

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Section 1: General

1.1 This standard covers the testing of metals subjected to tensile stresses for resistance to cracking failure in low-pH aqueous environments containing 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 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 suitability 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 This standard may be used for release or acceptance testing to ensure that the product meets a certain minimum level of EC resistance as prescribed in API(2) Specification 5CT,[3] ISO(3) 11960,[4] or as prescribed by the user or purchaser. This standard may also provide a quantitative measure of the product's EC resistance for research or informational purposes. This rating may be based on:

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

1.5 Safety Precautions: H_2S is an extremely toxic gas that must be handled with care. (See Appendix A [Nonmandatory]).

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(2) American Petroleum Institute (API), 1220 L St. NW, Washington, DC 20005-4070.

(3) International Organization for Standardization (ISO), Chemin de Blandonnet 8. Case Postale 401, 1214 Vermier, Geneva, Switzerland.

Section 2: Environmental Cracking Testing Variability

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

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

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

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

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

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

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

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

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

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

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

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

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

Section 3: Reagents

3.1 Reagent Purity

3.1.1 The test gases, sodium chloride (NaCl), acetic acid (CH_3COOH), sodium acetate (CH_3COONa), and solvents shall be reagent grade or chemically pure (99.5% minimum purity) chemicals. (See Appendix B [Nonmandatory]).

3.1.2 The test water shall be distilled or deionized and of quality equal to or greater than ASTM Type IV (ASTM D1193[6]). Tap water shall not be used.

3.2 Inert gas shall be used for removal of oxygen. Inert gas (nitrogen, argon, or other suitable nonreactive gases) shall be pre-purified (99.998% or greater).

Section 4: Test Specimens and Material Properties

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

4.2 When specified, tensile testing in accordance with standard test methods such as ASTM A370[7] shall be used to determine base material properties. Two or more test specimens shall be pulled, and the individual test results shall be averaged to determine the yield and ultimate strengths, percent elongation, and percent reduction in area for the material. Machining a tensile test specimen from material adjacent to and in the same position and orientation as the EC test specimen to be tested can minimize material property variations that normally occur from test specimen to test specimen.

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

4.4 When specified, hardness measurements shall be performed in accordance with ASTM E18 (Rockwell)[8] or ASTM E384 (Vickers)[9] before or after exposure to the test environment. These measurements shall not be made on the stressed evaluation portion of the test specimen. For test Methods A (subsize) and B, hardness shall be measured (1) on the test specimen, or (2) on an adjacent hardness test specimen that was sampled from a similar thickness/cross-section location as the material being tested. For test methods A (standard size), C, and D, hardness shall be measured on the test specimen. At least 3 indentations shall be performed on each specimen. Hardness test results shall be reported.

NOTE: When hardness testing on convex cylindrical surfaces, the results may not accurately indicate the true hardness. Corrections applied to the measured hardness shall be in accordance with the selected ASTM standard and reported.

Section 5: Test Vessels and Fixtures

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

5.2 Test vessels shall be capable of being purged to remove oxygen before beginning the test and of keeping air out during the test. Using a small outlet trap on the H_2S effluent line to maintain 250 Pa (0.036 psi) (1.0 inch in H_2O) back pressure on the test vessel may be used to prevent oxygen entry through small leaks or by diffusion up the vent line. (See Appendix B section titled “Reasons for Exclusion of Oxygen”).

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

5.4 Test vessels shall be constructed from materials that are suitable for the test environment. While some plastic test vessels and solution storage vessels give satisfactory service, others may cause varying test results from the time they are new until after they have been in continuous use. If plastic vessels are used, then the vessels shall be pre-conditioned with a documented procedure that has been validated. Glass test vessels have not exhibited this tendency and should not require pre-conditioning.

5.5 Test specimens shall be electrically isolated from test vessels and test fixtures made from dissimilar metals if the dissimilar metal is in contact with the test environment.

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

5.7 Galvanic Coupling

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

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

5.7.2 Particles of iron sulfide are 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 General

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

6.1.2 The test solution shall be maintained at 24 ± 3 °C (75 ± 5 °F) for Methods A, B, and C; and 24 ± 1.7 °C (75 ± 3.0 °F) for Method D. Also, the test temperature range shall be specified in accordance with testing at elevated temperature (see Section 7). Any variations beyond this range shall be reported.

6.1.3 The test solution used for each test method shall be reported.

6.1.4 If required, the concentration of H_2S in the test solution may be measured by iodometric titration or by other suitable methods. Accepted iodometric titration and reduced concentration of H_2S procedures are detailed in Appendix C (Nonmandatory).

6.1.5 Solutions and gas compositions other than those detailed below may also be used for fit-for-purpose testing and shall be reported.

6.1.6 The initial pH (before the introduction of the test gas) and final pH (at the end of the test) shall be measured and reported.

6.1.7 Unless expressly permitted in this standard, pH shall not be intentionally adjusted during the test.

6.1.8 Gas mixtures shall be certified pre-mixed or verified by suitable means, provided the pure component gases are individually certified.

6.1.9 The start of the test shall be measured from the time that saturation is achieved.

6.2 Test Solution A

6.2.1 Test Solution A is an acidified H_2S-saturated aqueous brine solution.

6.2.2 Test Solution A shall consist of 5.0 wt% sodium chloride and 0.5 wt% glacial acetic acid dissolved in distilled or deionized water (e.g., 50.0 g of NaCl and 5.0 g [4.8 mL] of CH_3COOH dissolved in 945 g of distilled or deionized water).

6.2.3 Test solution pH before contact with a test specimen and before H_2S saturation shall be between 2.6 and 2.8. Adjustment of test solutions chemistry to adjust pH is not allowed. If test solution pH is out-of-range then the solution shall be discarded. Each laboratory shall have a demonstrated and documented procedure for purging to verify that the pH has not exceeded 3.0 for Test Solution A after purging. During the test, pH may increase but shall not exceed 4.0. If the pH exceeds 4.0, the test is invalid. If the test-solution-volume to test-specimen-surface-area ratio is maintained and steps are taken to exclude oxygen from the test vessel as specified in this standard, the pH will not exceed this value.

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

6.3 Test Solution B

6.3.1 Test Solution B is an acidified and buffered H_2S-saturated aqueous brine solution.

6.3.2 Test Solution B shall consist of 5.0 wt% sodium chloride, 2.5 wt% glacial acetic acid, and 0.41 wt% sodium acetate dissolved in distilled or deionized water (e.g., 50.0 g of NaCl, 25.0 g [23.8 mL] of CH_3COOH, and 4.1 g of CH_3COONa dissolved in 921 g of distilled or deionized water).

6.3.3 Test solution pH before contact with a test specimen and before H_2S saturation shall be between 3.4 and 3.6. Adjustment of test solution chemistry to adjust pH is not allowed. If test solution pH is out- of-range, then the solution shall be discarded. During the test, pH may increase but shall not exceed 4.0. If the pH exceeds 4.0, the test is invalid. If the test-solution-volume to test-specimen-surface-area ratio is maintained and steps are taken to exclude oxygen from the test vessel as specified in this standard, the pH will not exceed this value.

6.3.4 Test Solution B may be used with carbon and low-alloy steels.

6.3.5 Test Solution B may be used in Methods A, C, and D.

6.4 Test Solution C

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

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

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

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

6.4.5 Test Solution C may be used with martensitic stainless steels.

6.4.6 Test Solution C may be used in Methods A, C, and D.

NOTE: The combination of a lower acetate concentration (0.4 g/L) and acidification with HCl rather than acetic acid leads to a significantly reduced concentration of both acetic acid and acetate ion in Test Solution C when compared with Test Solution B. Although this may be adequate for maintaining the bulk solution pH constant when testing corrosion-resistant alloys, the ability to resist changes of pH at the metal-solution interface is reduced. In cathodic regions, there is less acetic acid to counterbalance the tendency for an increased pH. In localized anodic regions, there is less acetate to restrain lowering of the pH due to metal ion hydrolysis. In the latter case, the lower acetate concentration associated with Test Solution C would give a more conservative result, in the sense that cracking could be more likely.

6.5 Test Solution D

6.5.1 Test Solution D is a buffered aqueous brine solution with a chloride content, H_2S partial pressure, and pH specified for mild sour conditions.

6.5.2 Test Solution D shall consist of 5 wt% sodium chloride and 0.40 wt% sodium acetate dissolved in distilled or deionized water (e.g., 50.0 g of NaCl and 4.0 g of CH_3COONa dissolved in 946 g of distilled or deionized water).

6.5.3 Hydrochloric acid (HCl) or sodium hydroxide (NaOH) shall be used to adjust the initial pH. The solution pH before contact with a test specimen and before H_2S saturation shall be between 3.8 and 4.0. During the test, pH may increase, but shall not exceed 4.6. If the pH exceeds 4.6, the test is invalid. If the test-solution-volume to test-specimen-surface-area ratio is maintained and steps are taken to exclude oxygen from the test vessel as specified in this standard, the pH will not exceed this value.

6.5.4 The test gas shall consist of 7.0 ± 0.3 mol.% H_2S with balance of nitrogen. After initial saturation, the gas should be bubbled continuously at a rate that maintains saturation.

6.5.5 Test Solution D may be used with high-strength steels such as API 5CT Grade C110.

6.5.6 Test Solution D may be used in Methods A, C, and D.

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

6.7 Deaeration Requirements and Procedures

6.7.1 Deaeration Requirements

The method of solution deaeration and solution transfer used for testing shall have the dissolved oxygen levels in the test solution (immediately prior to the introduction of the H_2S or H_2S mixed gas) of less than 50 ppb for low-alloy steels up to the strength level of 552 MPa (80 ksi) and 10 ppb for low-alloy steels above the strength level of 552 MPa (80 ksi) and corrosion resistant alloys. Each laboratory shall have a demonstrated and documented procedure to achieve the required dissolved oxygen levels.

6.7.2 Deaeration Procedures

6.7.2.1 The test solution shall be previously deaerated in a sealed vessel that is purged with inert gas at a rate of at least 100 mL/min per liter of test solution for at least one hour. The test vessel containing the test specimen(s) shall be purged separately with inert gas at a rate of at least 100 mL/min per liter of test solution for at least one hour. The deaerated test solution shall be transferred into the pre-purged test vessel, and it shall be further deaerated at a rate of at least 100 mL/min per liter of test solution for at least one hour or until the required dissolved oxygen level is reached.

6.7.2.2 Other methods of deaeration and transfer may be used if they have been demonstrated to achieve the required dissolved oxygen levels.

6.7.3 Oxygen contamination is evident by a cloudy (opaque) appearance of the test solution when the H_2S gas enters the test vessel. An opaque appearance of the test solution upon H_2S entry shall disqualify the test. The test specimen shall be removed and cleaned, and the test solution make-up, transfer, and deaeration procedure repeated.

Note: To achieve the required oxygen levels, consideration needs to be given to the following factors: type of tubing used, ingress of oxygen into the test cell during the test, etc.

Section 7: Testing at Elevated Temperature/Pressure

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

7.2 Testing at elevated temperatures and pressures involves additional safety considerations compared to room temperature and atmospheric pressure testing. While some general guidance is given herein, it may not address all aspects and should be supplemented to comply 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 shall be demonstrated, by measuring H_2S concentration in either the test solution or gas phase, that the required test conditions have been maintained. This information must be reported with the test data.

7.3 Test Equipment

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

7.3.1 The test vessel shall be equipped with a thermocouple well or other means of measuring the temperature of the test solution, inlet and outlet ports for gas, a dip tube on the inlet port, and a pressure-measuring device.

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

7.3.3 A bursting (rupture) disc or pressure-relief valve should be 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.

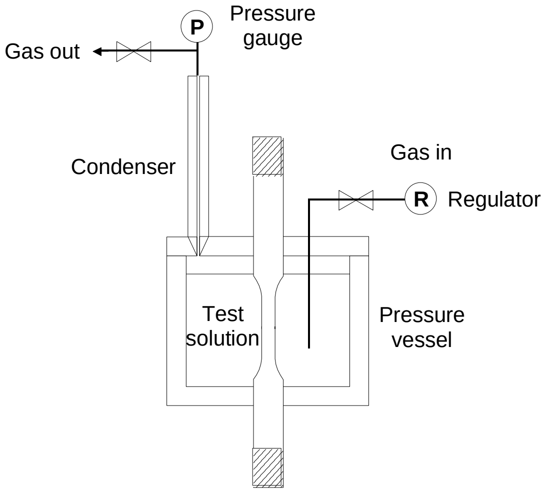


Figure 1: Schematic Arrangement of Test Equipment for Method A—NACE Standard Tensile Test

7.4 Test Solution

The test solution used in the test may be selected as required by the test specification. The test solution usually consists of brine (NaCl) at concentrations up to saturation. Buffered acidification is permitted, analogous to room-temperature methods. Also, the test solution may contain elemental sulfur.[10,11,12]

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.[13] The test gas mixture should be contained in a standard gas bottle equipped with a suitable pressure regulator (usually stainless steel) capable of gas delivery to the total test pressure required. A commercially supplied gas mixture with composition determined by analysis should be used.

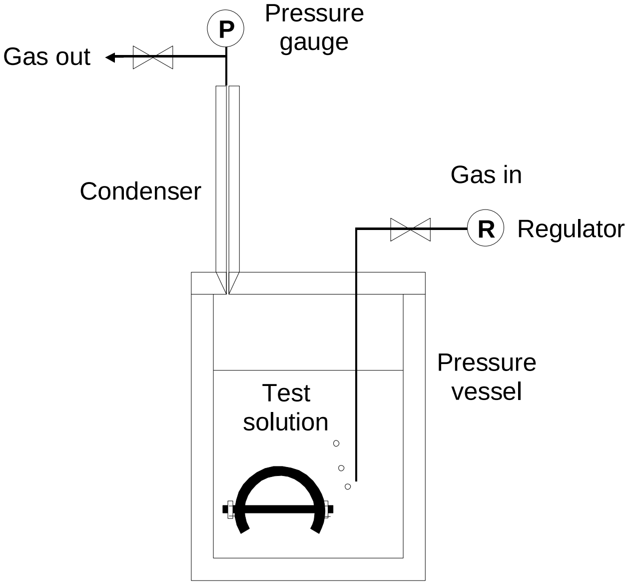


Figure 2: Schematic Arrangement of Test Equipment for Method B—NACE Standard Bent-Beam Test, Method C—NACE Standard C-Ring Test, and Method D—NACE Standard Double-Cantilever-Beam Test

7.6 Test Stresses

The test stresses may be derived from the actual yield strength at ambient temperature or from the actual yield strength at the planned test temperature or as otherwise required by the test specification.

7.7 Test Procedure

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

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

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

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

7.7.4 The H_2S partial pressure, ppH_2S, in the test environment shall be determined by one of the following two methods:

7.7.4.1 Test vessel heated before test gas admitted

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

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

7.7.4.1.3 The H_2S partial pressure, ppH_2S, in the test environment is approximated by Equation (1):

$$ppH_2S = (P_T - P_1)\,(X_{H2S}) \tag{1}$$

where:

P_T = total absolute test pressure;
P_1 = vapor pressure above the test solution; and
X_{H2S}= mole fraction of H_2S in the test gas.

7.7.4.2 Test gas admitted before test vessel heated.

Test gas may be admitted to the test vessel before heating if a proven means of calculating ppH_2S can be demonstrated.

7.7.4.3 The test method used shall be reported with the test data.

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

7.7.6 The test duration shall be as specified for the applicable test method (A, B, C, or D). The test temperature for Methods A, B, and C shall be maintained within ±3 °C (±5 °F) of the specified test temperature and recorded manually on a daily basis or at shorter intervals by data recorder. For Method D, test temperature shall be maintained within ±1.7 °C (±3.0 °F). Pressure shall be monitored and recorded daily. If test pressure falls by more than 40 kPa (6 psi) below the required test pressure, the test gas must be replenished.

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

Section 8: Method A—NACE Standard Tensile Test

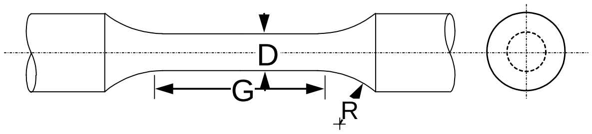
8.1 Method A, the NACE standard tensile test, provides for evaluating metals for EC resistance under uniaxial tensile loading. It offers a simple unnotched test specimen with a well-defined stress state. EC susceptibility with Method A is usually determined by time-to-failure. Tensile test specimens loaded to a particular stress level give a failure/no-failure test result. When multiple test specimens are tested at varying stress levels, an apparent threshold stress for EC can be obtained.[14]

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

8.2 Test Specimen

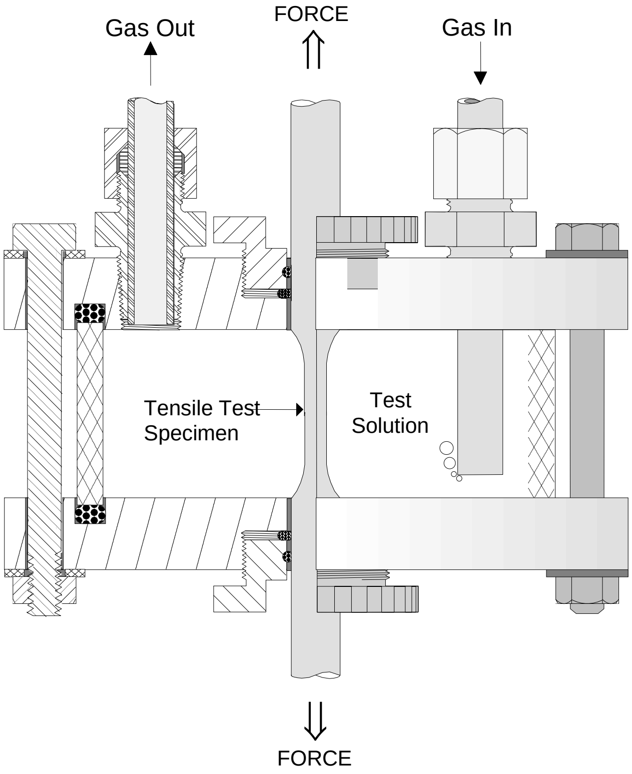
8.2.1 The size and shape of the material available for testing often restricts selection of test specimens. The orientation of the test specimen can affect the results and should be noted.

8.2.2 The gauge section of the standard tensile test specimen (see Figure 3[a]) shall be 6.35 ± 0.13 mm (0.250 ± 0.005 in) in diameter by 25.4 mm (1.00 in) long (see ASTM A370). A subsize tensile 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, tensile test specimens should be stored in a low-humidity area, in a desiccator, or in uninhibited oil until ready for testing.



Dimension	**Standard tensile test specimen**	**Subsize tensile test specimen**
D	6.35 ± 0.13 mm (0.250 ± 0.005 in)	3.81 ± 0.05 mm (0.150 ± 0.002 in)
G	25.4 mm (1.00 in)	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

8.2.3 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.

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

(1) Eliminate undercutting of fillet radii in machined test specimens; and

(2) Machine the test specimen gauge section with a slight (0.05 to 0.13 mm [0.002 to 0.005 in]) taper that produces a minimum cross-section in the middle of the gauge section.

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

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

8.2.6 For all materials, the average surface roughness of the gauge section shall be 0.25 µm (10 µin) or finer, as defined by Ra value in ISO 4287.[15] Final surface finish may be obtained by mechanical polishing or electropolishing if the roughness requirement is met. The finishing processes shall be reported with the test data. When electropolishing, bath conditions must be such that the test specimen does not absorb hydrogen during the procedure. When agreed with the end user, electropolishing shall only be used with low-alloy steels having a maximum of 1.5 weight percent chromium level. Each laboratory shall have a demonstrated and documented procedure for electropolishing and validating that electropolishing has comparable results to surface ground/mechanically polished specimens (per test material, grade and test condition).

8.2.7 When a standard tensile test specimen cannot be obtained from the material because of its size or shape, a subsize tensile test specimen may be used. However, subsize tensile test specimens can produce shorter failure times than those observed for standard tensile test specimens. The report of test data using subsize tensile test specimens shall clearly state the use of subsize test specimens. If an alternate specimen configuration (one not specified in this document) is used, the dimensions shall be clearly stated in the test report.

8.2.8 Test Specimen Identification

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

8.2.9 Test Specimen Cleaning

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

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

8.3 Test Solutions—see Section 6.

8.4 Test Equipment

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

8.4.2 Tensile tests should be performed with constant-load or sustained-load (proof-ring or spring-loaded) devices (see ASTM G49).[16]

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 and maintained to minimize bending and torsional loads.

Note: Unwanted bending stress increases the maximum total stress and may lead to an over-conservative test, specimen failure, and rejection of a material which otherwise would pass the Method A test if bending was absent or minimized.

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

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

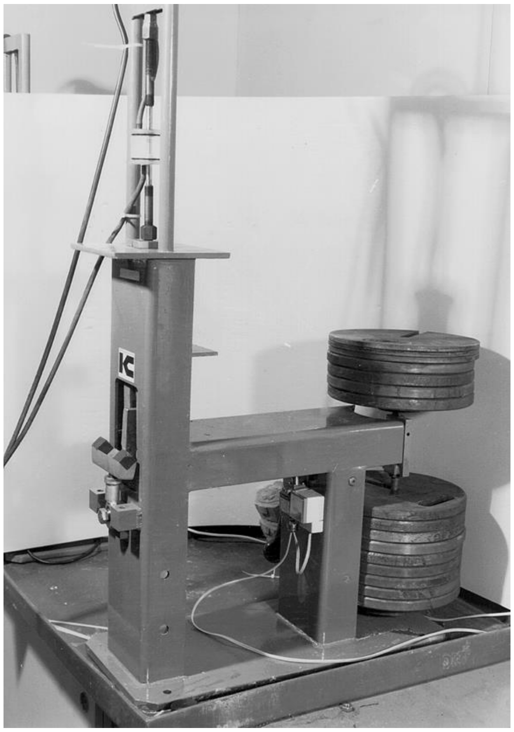
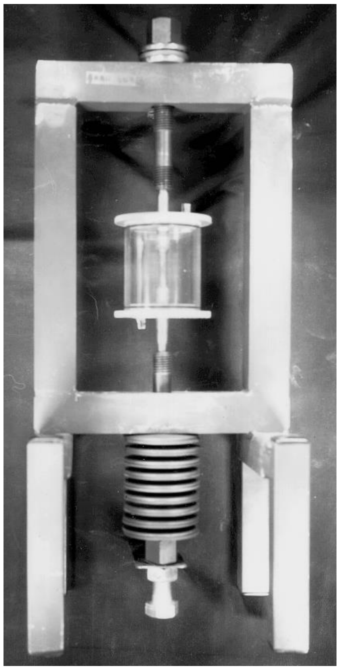


Figure 4: Constant-Load (Dead-Weight) Device

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

(a) Proof ring



(**b) Spring-loaded**

Figure 5: Sustained-Load Devices

8.4.5.1 If 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 test specimen shall lie within the load range of the proof ring. Accordingly, proof rings shall be selected so that the applied load produces 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.

The proof ring deflection should be measured during the test or at the test completion.

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 test specimen with negligible friction.

8.4.6.2 In cases in which the complete test fixture is 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 test solution volume of 30 ± 10 mL/cm^2 of test specimen surface area.

8.5 Stress Calculations

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

$$P = S \times A \tag{2}$$

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 tensile test specimen shall be measured, and the tensile 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. 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 Deaeration requirements are given in Paragraph 6.7.

8.6.6 The test solution shall then be saturated with H_2S at a minimum flow rate per a documented procedure that has been validated to obtain 2,300 mg/L minimum H_2S concentration or other minimum value H_2S concentration that is proportional to less than 1 bar H_2S partial pressures (at the extent of the test specimen locations). When Solution D is used, the minimum H_2S concentration shall be 160 mg/L. Saturation shall occur within an hour of contact with specimens in test vessels up to 1 L (see Note 1). For larger test vessels, saturation can require more than two hours. Analysis shall be done using iodometric titration (See Appendix C or other suitable methods). The validation of the documented procedure shall be performed after saturation at the start of test, after 24 hours, weekly and at the end of the test. A continuous flow of H_2S through the test vessel and outlet trap shall be maintained with a positive pressure of H_2S throughout the test that prevents air from entering the test vessel.

NOTE 1: One method found to give saturation within one hour in a proof ring test (approximately 1/3 L) is to purge at 100 mL per minute for 60 minutes.

NOTE 2: Laboratories at high elevations may find it necessary to compensate for lower atmospheric pressure in order to achieve the required saturation levels.

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

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

8.7 Failure Detection

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

8.7.1 For all materials, failure is either:

(a) Complete separation of the tensile test specimen; or

(b) Visual observation of cracks on the gauge section of the tensile test specimen at 10X after completing the 720 hour test duration. Investigative techniques employing metallography, scanning electron 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 tensile test specimen passes the test.

8.7.2 Time-to-failure may 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 (see Table 1).

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

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

Table 1

NACE Uniform Material Testing Report Form (Part 1): Testing in Accordance with NACE Standard TM0177[A] Method A—NACE Standard 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.
[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 (continued)
NACE Uniform Material Testing Report Form (Part 2): Testing in Accordance with NACE Standard TM0177 Method A—NACE Standard Tensile Test

Lab Data for Material: __________________________Tested per NACE Standard TM0177(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 ☐ Finishing Process____________________

Chemistry: ☐ Test Solution A ☐ Test Solution B ☐ Test Solution C (define)__________ ☐ Test Solution D ☐ Other Test Solution_____

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

For elevated temperature tests: ☐ Test vessel heated before test gas ☐ Test gas admitted before test vessel heated

Material Identification	**Test Specimen Properties**							**Test Stress (% of Yield Strength)**								**Test 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 Standard TM0177.

(B) Location of test specimen 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. Consideration may be given to the use of the service temperature when determining the value of the yield strength used in the calculations of the test stresses to be applied.

(E) Enter pH for test conducted on nonfailed tensile test specimen at highest stress if summarizing data.

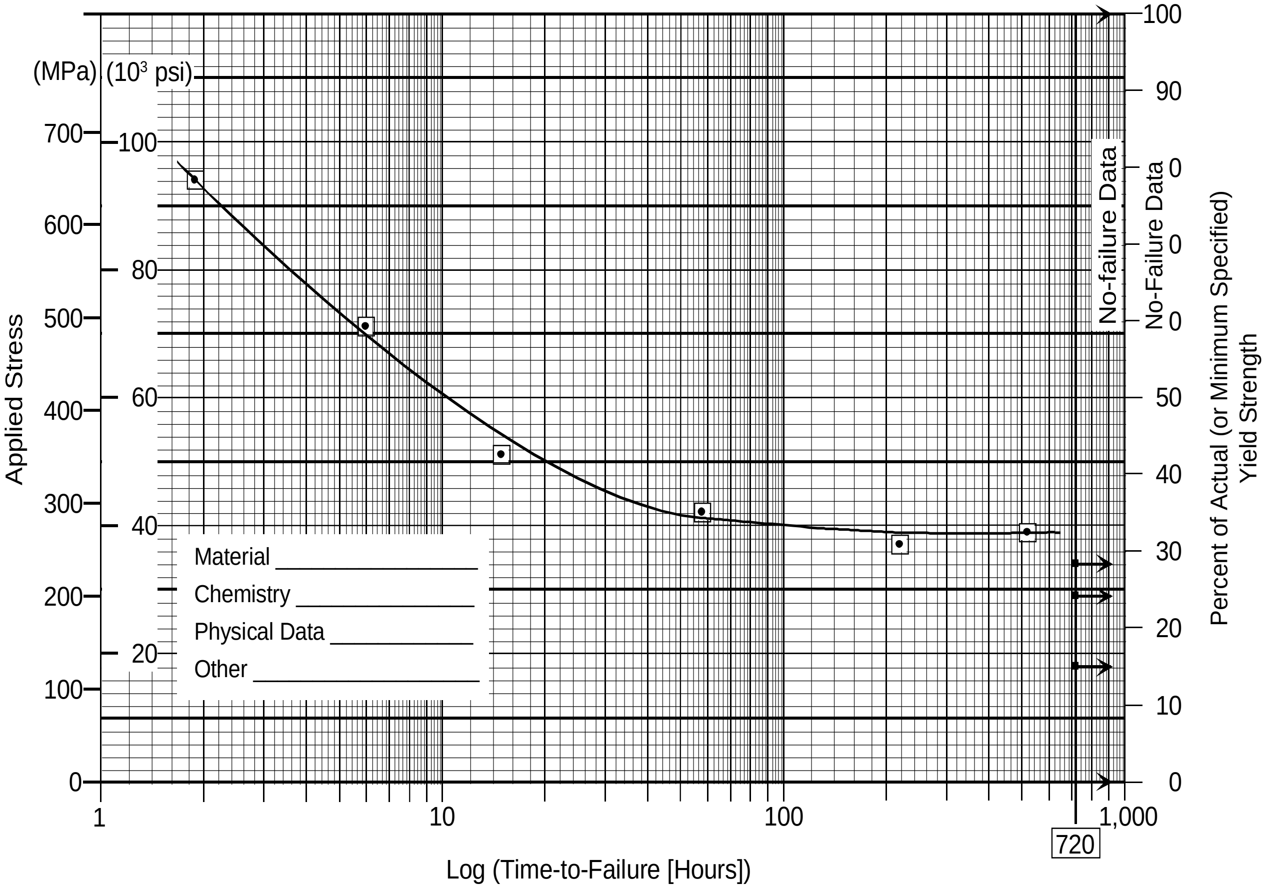


Figure 6: Applied Stress vs. Log (Time-to-Failure)

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 statistically based critical stress factor (S_c) for a 50% probability of failure 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.[17]

9.1.1 This section sets forth the procedure for bent-beam testing at room temperature and atmospheric pressure. Special considerations for testing at elevated temperature and pressure are set forth 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 different bending stress. The stressed test specimens then are exposed to the test environment, and the failure (or no-failure) by cracking is determined. From these data obtained by testing multiple test specimens at varying deflections, a statistically based S_c for a 50% probability of failure is calculated to indicate the material's resistance to SSC.

9.1.2.2 This method constitutes a constant-deflection test of high 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

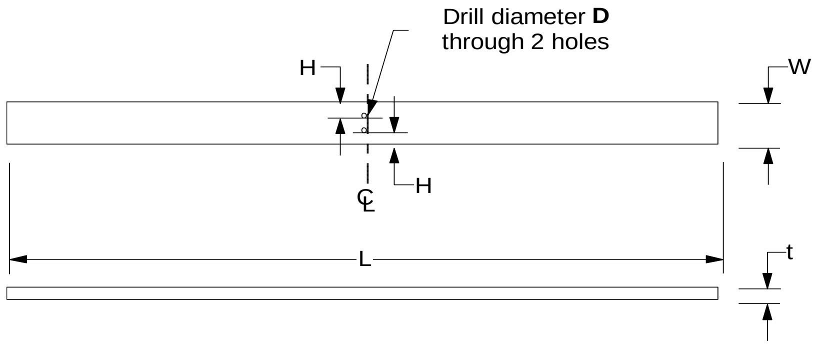
9.2.1 The dimensions of the standard bent-beam test specimen shall be 4.57 ± 0.13 mm (0.180 ± 0.0050 in) wide, 1.52 ± 0.13 mm (0.060 ± 0.0050 in) thick, and 67.3 ± 1.3 mm (2.65 ± 0.050 in) long (see Figure 7). After machining, test specimens shall be stored in a low-humidity area, in a desiccator, or 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 shall 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) shall be drilled at the midlength of the test specimen, centered 1.58 mm (0.0620 in) from each side edge. Holes shall be drilled before machining the final surface.



Dimension	Size	
	(mm)	(in)
L	67.3 ± 1.3	2.65 ± 0.050
t	1.52 ± 0.13	0.060 ± 0.0050
W	4.57 ± 0.13	0.180 ± 0.0050
H	1.58 ± 0.05	0.062 ± 0.002
D	0.71 ± 0.0013	0.028 ± 0.0005 (No. 70 Drill)

Figure 7: Dimensional Drawing of the Standard Bent-Beam Test Specimen

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.

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

9.3 Test Solution

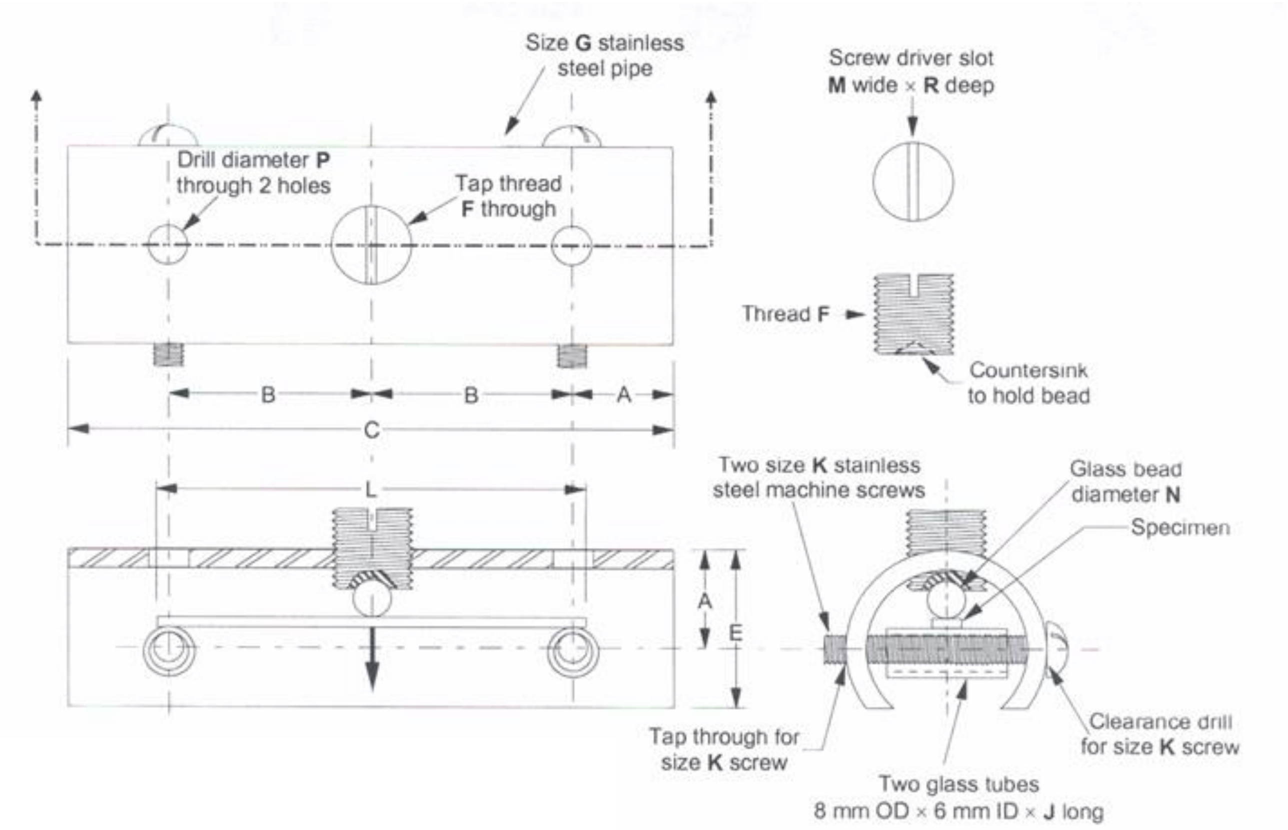
9.3.1 The test solution shall consist of 0.5 wt% glacial acetic acid dissolved in distilled or deionized water (e.g., 5.0 g [4.8 mL] of CH_3COOH dissolved in 995 g of distilled or deionized water). NaCl shall not be added to the test solution.

9.3.2 Use of Test Solutions A, B, C, and D 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.

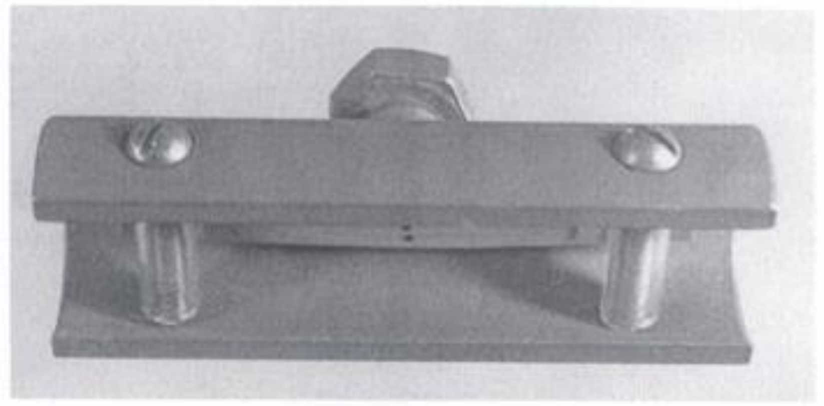
9.4.2 Tests shall be performed using constant-deflection fixtures that employ three-point bending of the test specimen. (See Figure 8).



Dimension	Size	
	(mm)	(in)
A	15.9	0.625
B	31.75 ± 1.27	1.250 ± 0.050
C	95.3	3.75
E	25.4	1
F(A)	M12 x 1.25	0.5-20 NF thread
G(A)	25.4	1
J	19.1	0.75
K(A)	M6 x 40 long	#10-32 NF x 1.5 long
L	≥ 66.70	≥ 2.625
M	1.6	0.063
N(A)	6.35	0.25
P	6.35	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

9.4.3 Test fixtures immersed in a test solution should resist general corrosion (UNS(4) 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 shall be sized to maintain a test solution volume of 30 ± 10 mL/cm^2 of test specimen surface area. Maximum volume of the test vessel should be 10 L.

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

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

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(4) Unified Numbering System for Metals and Alloys (UNS). UNS numbers are listed in *Metals & Alloys in the Unified Numbering System* (latest revision). (Warrendale, PA: SAE International and West Conshohocken, PA: ASTM International).

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

$$D = \frac{S\ell^2}{6Et} \tag{3}$$

where:

D = deflection;
S = nominal outer fiber pseudo-stress;
ℓ = distance between centerlines of end supports;
E = elastic modulus; and
t = thickness of test specimen.

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 a dial or digital gauge and fixture. 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 Deaeration requirements are given in Paragraph 6.7.

9.6.5 The test solution shall then be saturated with H_2S at a rate of at least 100 mL/min for at least 20 min/L of test solution. The H_2S in the test vessel shall be replenished periodically by bubbling H_2S for a duration of 20 to 30 min at a rate of at least 100 mL/min/L of test 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 work week.

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

9.6.7 Additional test specimens and iterative testing may be necessary to define the S_c 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 shall be made. 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 nominal outer fiber pseudo-stress (S) values shall be reported. Time-to-failure data are optional.

9.8.2 The S_c shall be calculated using Equation (4) for S values expressed in MPa, or Equation (5) for S values expressed in psi:

$$S_c = \frac{\frac{\sum S}{68.95\ \text{MPa}} + 2\sum T}{n} \tag{4}$$

where:

S = nominal outer fiber pseudo-stress (in MPa) 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.

When Equation (4) is used, all pseudo-stress data that are more than ±210 MPa from the initial calculated value S_c x 68.95 MPa shall be discarded, and a new S_c value shall be recalculated. The recalculated S_c value eliminates low and high bias data.

$$S_c = \frac{\frac{\sum S}{10^4\ \text{psi}} + 2\sum T}{n} \tag{5}$$

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.

When using Equation (5), all pseudo-stress data that are more than ±3.0 x 10^4 psi from the initial calculated value S_c x 10^4 psi shall be discarded, and a new S_c value shall be recalculated. The recalculated S_c value eliminates low and high bias data.

9.8.3 The calculated S_c value for each material tested shall be reported. If S_c is recalculated, the recalculated S_c value shall be reported, not the initial calculated S_c value.

9.8.4 If known, the chemical compositions, heat treatment, mechanical properties, and other data taken shall be reported.

9.8.5 Table 2 shows the recommended format for reporting the data.

Table 2
NACE Uniform Material Testing Report Form (Part 1): Testing in Accordance with NACE Standard TM0177(A) Method B—NACE Standard 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.

(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 (continued)
NACE Uniform Material Testing Report Form (Part 2): Testing in Accordance with NACE Standard TM0177 Method B—NACE Standard Bent-Beam Test

Lab Data for Material: ____Tested per NACE Standard TM0177(A)

Test Specimen Geometry: ☐ Standard ☐ Nonstandard _____________ Nominal Size ______________ Length ______________________
☐ Statistical S_c Method Applied _____________________________

Chemistry: ☐ 0.5 wt% glacial acetic acid in distilled or deionized water ☐ 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							Psuedo-Stress (S) Value ( )								S_c Value	Test 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 Standard TM0177.

(B) Location of test specimen 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 bent-beam test specimen at highest stress if summarizing 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. 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 C-ring test specimens at varying stress levels, an apparent threshold stress for EC can be obtained.

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

10.2 Test Specimen

10.2.1 An unnotched C-ring test specimen in accordance with ASTM G38[18] shall be used. Sizes for C-rings may vary over a wide range, but C-rings with an outside diameter (OD) of less than about 15.9 mm (0.625 in) should not be used 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 C-ring test specimen or be electrically isolated from the C-ring 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 shall be stored in a low-humidity area, 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 shall be reported with the test data.

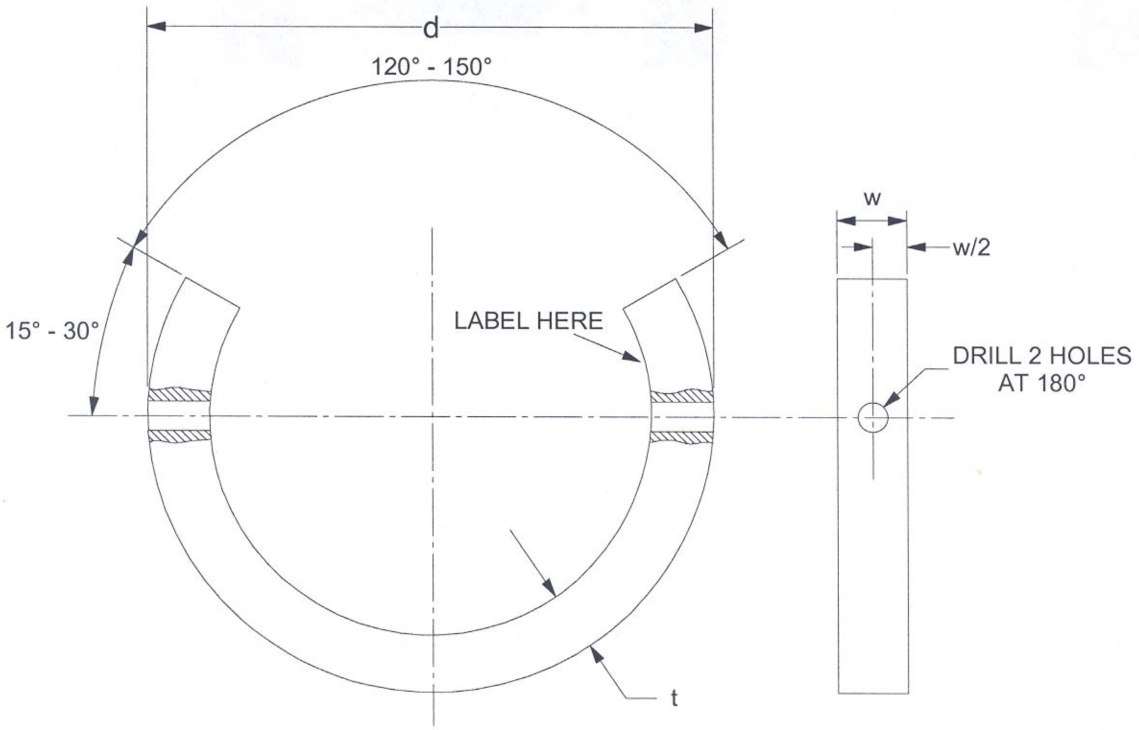


Figure 9: Dimensional Drawing of the C-Ring Test Specimen

10.2.5 Test Specimen Identification

The C-ring test specimen end segments may be stamped or vibratory stenciled.

10.2.6 Test Specimen Cleaning

10.2.6.1 Before testing, C-ring test specimens shall be degreased with solvent and rinsed with acetone.

10.2.6.2 The C-ring test specimen shall not be contaminated after cleaning. Test specimens should be handled with new disposable gloves. Powdered gloves shall be cleaned of powder prior to handling test specimens.

10.3 Test Solutions—see Section 6.

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

10.4.2 The C-ring test specimen shall be so supported that nothing except the test solution contacts the stressed area.

10.4.2.1 The supporting fixture shall be constructed of material compatible with the test solution.

10.4.2.2 Galvanic effects between the C-ring test specimens, supporting fixtures, and test vessel shall be avoided. For example, an isolating bushing or washer may 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 test solution volume of 30 ± 10 mL/cm^2 of test specimen surface area.

10.4.3.2 A fritted glass bubbler should be used to introduce the inert gas and H_2S below the array of C-ring test specimens. The bubbles should not impinge on the C-ring 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 (6):

$$D = \frac{\pi d\ (d\text{-}t)\ S}{4tE} \tag{6}$$

where:

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

10.5.1.1 Deflections calculated by Equation (6) 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 (6) may be used for carbon and low-alloy steels 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.

10.5.1.3 No equation exists to calculate the deflection needed to stress C-ring 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 test 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 C-ring 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).

Deflections shall be measured at the center line 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 C-ring test specimens shall be cleaned and placed into the test vessel.

10.6.4 Deaeration requirements are given in Paragraph 6.7.

10.6.5 The test solution shall then be saturated with H_2S at a minimum flow rate per a documented procedure that has been validated to obtain 2,300 mg/L minimum H_2S concentration or other minimum value H_2S concentration that is proportional to less than 1 bar H_2S partial pressures (at the extent of the test specimen locations). When Solution D is used, the minimum H_2S concentration shall be 160 mg/L. Saturation shall occur within an hour of contact with specimens. For larger test vessels, saturation can require more than two hours. Analysis shall be done using iodometric titration (See Appendix C or other suitable methods). The validation of the documented procedure shall be performed after saturation at the start of test, after 24 hours, weekly, and at the end of the test. A continuous flow of H_2S through the test vessel and outlet trap shall be maintained with a positive pressure of H_2S throughout the test that prevents air from entering the test vessel.

NOTE: Laboratories at high elevations may find it necessary to compensate for lower atmospheric pressure in order to achieve the required saturation levels.

10.6.6 The termination of the test shall be at all C-ring test specimens' failure or after 720 hours, 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 for each specimen. If time-to-failure data are recorded, they shall be reported.

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

10.8.3 Table 3 shows the recommended format for reporting the data.

Table 3
NACE Uniform Material Testing Report Form (Part 1): Testing in Accordance with NACE Standard TM0177[A] Method C—NACE Standard 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.
[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 (continued)
NACE Uniform Material Testing Report Form (Part 2): Testing in Accordance with NACE Standard TM0177 Method C—NACE Standard C-Ring Test

Lab Data for Material: ____________________ **Tested per NACE Standard TM0177(A)**

Test Specimen Geometry: **Outside Diameter** _____ **Wall/Thickness** _______ **Width** ______________
Test Equipment: ☐ **Bolting Material Same as Specimen**
☐ **Correction for Yield Applied**

Chemistry: Solution ☐ **Test Solution A** ☐ **Test Solution B** ☐ **Test Solution C (define)**__________ ☐ **Test Solution D** ☐ **Other Test** ______________________

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

Material Identification	**Test Specimen Properties**							**Applied Stress (% of Yield Strength)**								**Test 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 Standard TM0177.

(B)Location of test specimen 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 C-ring test specimen at highest stress if summarizing data.

Section 11: Method D—NACE Standard Double Cantilever Beam 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.[19] The subject of fracture mechanics testing for evaluation of EC resistance is currently under consideration by NACE TG 085 and Work Group (WG) 085c, and ASTM Committees E8.06.02 and G1.06.04. 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 K_{ISSC} is not an intrinsic material property, but depends on the environmental exposure conditions and method of testing. Nevertheless, the values obtained by carefully adopting this standard can be used for comparative purposes.

11.1.2 This section sets forth the procedure for DCB testing at room temperature and atmospheric pressure and enables computation of K_{ISSC}. When the special considerations set forth 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 set forth in Paragraph 11.6 for K_{ISSC}. 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 DCB test specimen design shall be in accordance with Figure 11(a). A double-tapered wedge shall be used to load the DCB test specimen (see Figure 11 [b]). The double-tapered wedge shall be made of the same material as the DCB test specimen or of the same class of material as the DCB 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 section. After machining, test specimens and wedges shall be stored in a low-humidity area, in a desiccator, or in uninhibited oil until ready for testing.

11.2.2 The standard DCB test specimen thickness shall be nominally 9.53 mm (0.375 in); complete dimensions are shown in Figure 11(a). The surface roughness of the test specimen shall not exceed 0.81 µm (32 µin) finish. When the material being tested is too thin to meet this requirement, optional thicknesses as noted in Figure 11(a) may be considered. Subsize DCB test specimens of some carbon and low-alloy steels may give lower K_{ISSC} values than the standard DCB test specimens.

11.2.3 Full-thickness DCB 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 (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 or temper the material.

11.2.5 If testing materials of low K_{ISSC} (below 22 to 27 MPa√m [20 to 25 ksi√in]) or materials in which crack initiation is difficult, e.g., lower-yield-strength materials, introducing the electrodischarge-machined (EDM) slot noted in Figure 11(a) or a fatigue precrack is very helpful in avoiding sidearm cracking and in initiating SSC, respectively. Fatigue precracking of the DCB specimen should be conducted under load control at a convenient frequency. The acceptable range for a fatigue precrack is 1 to 3 mm (0.04 to 0.12 in) past the base of the chevron or end of the EDM slot. To avoid erroneous high results, the maximum precracking load shall be the lesser of 70% of the expected initial K_I imparted by the wedge or 30 MPa√m (27 ksi√in). The ratio of minimum to maximum load shall be in the range of 0.1 to 0.2. The precrack should be sharpened at two-thirds of the maximum precracking load for approximately 20,000 cycles to extend the crack through any zone of residual compressive stresses that might have developed.

11.2.6 Test Specimen Identification

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

NOTE: To maintain identification during machining, the specimen should initially be marked on the end that is not wedge loaded.

11.2.7 Hardness Measurement

When specified, three or more hardness readings shall be taken on each test specimen. The average value and face tested shall be reported with the test data. The hardness should be measured within the final 50 mm (2 in) of length on the long narrow faces of standard size and 6.35 mm (0.250 in) thick specimens, (see Figure 10) or on the broad faces near the side groove of thinner subsized specimens.

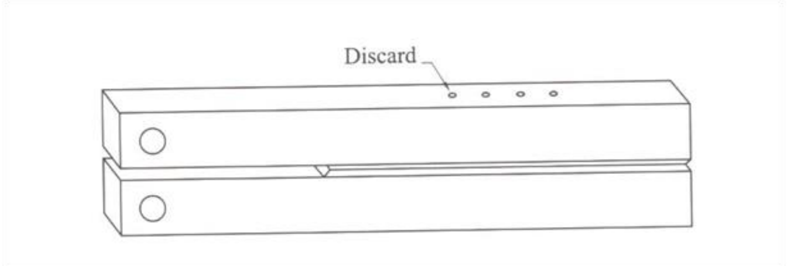


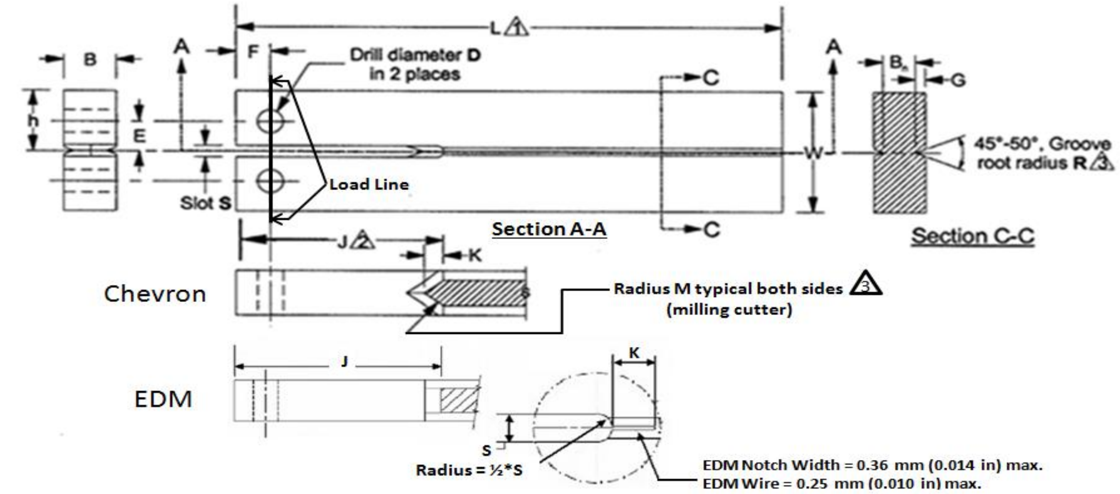
Figure 10: Location of Hardness Impressions on DCB Specimen

11.2.8 Dimensional Check

Dimensions B, B_n, 2h, and the distance of the hole centers from the near end of the DCB test specimen shall be measured. (A blade micrometer should be used for measuring B_n). Any values that lie outside the limits shown in Figure 11 (a) shall be recorded for later use in computing K_{ISSC} (see Paragraph 11.6.3).

11.2.9 Test Specimen Cleaning

Test specimens shall be degreased with solvent and rinsed in acetone. Test specimen degreasing should take place before fatigue precracking and wedge insertion. All degreased test specimens shall be handled in such a manner to avoid contamination. All degreased test specimens should be handled with new powder-free disposable gloves. If used, powdered gloves shall be cleaned of powder prior to handling DCB test specimens.



NOTES:

1 A DCB specimen of length U may be used if very low cracking resistance is expected.

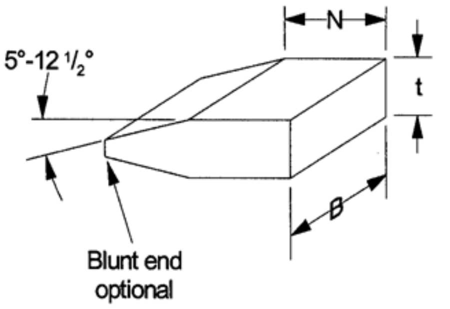
2 A short electrodischarge-machine (EDM) notch may substitute the Chevron crack starter.

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	6.35 + 0.25/–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 ± 0.76	1.500 ± 0.030
K	3.18 ± 0.25	0.125 ± 0.010
L	101.60 ± 1.60	4.000 ± 0.0625
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	2.39 ± 0.05	0.09375 ± 0.002
U	127	5
W	25.40 ± 0.05	1.000 ± 0.002
Z	± 0.05	± 0.002

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

Figure 11(a): Dimensional Drawing of the DCB Specimen



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

Figure 11(b): DCB Specimen—Double-Tapered Wedge

11.3 Test Solutions—see Section 6.

11.4 Test Equipment

11.4.1 The test vessel shall be sized to maintain a test solution volume of 10-12 mL/cm^2 of test specimen area (generally, 1 L of test solution per DCB specimen is used). The maximum volume of the test vessel should be 10 L for ease of test solution saturation.

11.4.2 A slotted base plate or other test specimen holder (electrically isolating) should be used to ensure uniform spacing and orientation of the DCB 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 DCB test specimen. Alternatively, the arms may be spread by a tensile testing machine. Care shall be taken to avoid over spreading.

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

11.5 Testing Sequence

11.5.1 Clean the DCB test specimens as indicated in Paragraph 11.2.9.

11.5.2 Select wedges to achieve specified arm displacement.

11.5.2.1 Measure the slot thickness.

11.5.2.2 The arm displacement shall be carefully controlled because initial stress intensity affects the final K_{ISSC}.[20] The arm displacement shall not exceed the limits listed in Tables 4 and 5. The acceptable arm displacements given in Table 4 may provide guidance in the selection of arm displacements for testing other grades of carbon and low-alloy steels. In a similar way, Table 6 provides guidance in choosing arm displacements for low-alloy steel products not listed in Table 4 and for other alloys.

NOTE: For nonstandard products and fit-for-purpose testing, some experimentation may be necessary to determine the required arm displacement range, i.e., one causing crack growth of about 12.7 to 25.4 mm (0.5 to 1.0 in).

11.5.2.2.1 When testing steels with yield strengths below 550 MPa (80 ksi), the arm displacement (δ) may be computed using Equation (7a) or (7b), in which YS is yield strength:

$$\delta\,(\text{mm}) = \left[2.02 - \frac{\text{YS (MPa)}}{530}\right] \pm 0.13\ \text{mm} \tag{7a}$$

$$\delta\,(\text{in}) = \left[0.080 - \frac{\text{YS (ksi)}}{1{,}953}\right] \pm 0.0050\ \text{in} \tag{7b}$$

11.5.2.2.2 For tests in Solution A and 100% H_2S, the arm displacement range is listed in Tables 4 and 6.

11.5.2.2.3 For tests in Solution D and 7% H_2S, the arm displacement range for API 5CT Grade C110 is listed in Table 5.

Table 4
Arm Displacements for API and Other Grade Oilfield Tubular Steels in Solution A and 100%H_2S

Grade[(A)]	Yield Strength Range		Acceptable Arm Displacement (δ)	
	MPa	**(ksi)**	**mm**	**(0.001 in)**
L80	552-655	(80-95)	0.71-0.97	(28-38)
C90	621-724	(90-105)	0.71-0.79	(28-31)
T95	655-758	(95-110)	0.66-0.74	(26-29)
R95	655-758	(95-110)	0.66-0.74	(26-29)
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)
C-110	758-827	(110-120)	0.46-0.54	(18-21)
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
Arm Displacement for API 5CT Grade C110 in Solution D and 7% H_2S

Grade(A)	Yield Strength Range		Acceptable Arm Displacement (δ)	
	MPa	(ksi)	mm	(0.001 in)
C110	758-827	(110-120)	0.84-0.92	(33-36)

(A) API grades unless noted otherwise.

Table 6
Suggested Arm Displacements for Selected Alloys and Strength Levels in Solution A and 100% H_2S

Material Yield Strength		Arm Displacement (δ), mm (0.001 in.)(A)									
MPa	(ksi)	Low-Alloy Steels(B)		UNS J91540		Duplex Stainless Steels		UNS N10276		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.

11.5.2.2.4 On both sides of the test specimen, measure the initial spacing of the loading holes in the DCB test specimen for use in determining actual arm displacement at the load line. Initial spacing may be measured using lines inscribed vertically along the load line of each side of the test specimen (see Figure 11[a]). Other methods of determining the actual arm displacement may be used if they have been demonstrated to achieve the required accuracy. The average of the dimensional measurements taken from each side shall be used as the initial spacing value.

11.5.2.2.5 Select a wedge thickness that would produce an expected arm displacement in the desired range.

NOTE: For materials with high cracking resistance or testing in a higher-pH environment, fatigue precracking and/or higher initial arm displacements (δ) should be used.

11.5.2.2.6 Press the wedge into the slot and flush with the end of the DCB test specimen.

11.5.2.2.7 On both sides of the test specimen, re-measure the spacing of the holes at the load line, use the average of the dimensional measurements taken from each side as the post-wedge insertion spacing, and then compute the actual arm displacement (δ). Re-measurement may be performed using the same inscribed line described in Paragraph 11.5.2.2.4 above. For the SSC-resistant carbon and low-alloy steels in Table 6, 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 DCB test specimen.

11.5.3 Degrease the DCB test specimens with solvent and rinse in acetone. All degreased test specimens shall be handled in such a manner as to avoid contamination. All degreased test specimens should be handled with new powder-free disposable gloves. If used, powdered gloves shall be cleaned of powder prior to handling DCB test specimens.

NOTE: Fatigue precracked DCB test specimens shall be cleaned by wiping, not by using immersion.

11.5.4 Place the DCB test specimens in the test vessel. The test specimen shall be exposed to H_2S within 24 hours of wedge insertion.

11.5.5 Deaeration requirements are given in Paragraph 6.7.

11.5.6 The test solution shall then be saturated with H_2S at a minimum flow rate per a documented procedure that has been validated to obtain 2,300 mg/L minimum H_2S concentration or other minimum value H_2S concentration that is proportional to less than 1 bar H_2S partial pressures (at the extent of the test specimen locations). When Solution D is used, the minimum H_2S concentration shall be 160 mg/L. Saturation shall occur within two hours of contact with specimens. Analysis shall be done using iodometric titration (See Appendix C or other suitable methods). The validation of the documented procedure shall be performed after saturation at the start of test, after 24 hours, weekly, and at the end of the test. A continuous flow of H_2S through the test vessel and outlet trap shall be maintained with a positive pressure of H_2S throughout for the duration of the test that prevents air from entering the test vessel.

NOTE: Laboratories at high elevations may find it necessary to compensate for lower atmospheric pressure in order to achieve the required saturation levels.

11.5.7 The temperature of the test solution shall be maintained within the range of 24 ± 1.7 °C (75 ± 3.0 °F) throughout the test because K_{ISSC} values for low-alloy steels have been found to vary significantly with temperature in the vicinity of room temperature. Temperature of the test solution shall be monitored, and the average reported.

11.5.8 The test duration for carbon and low-alloy steels shall be at least 14 days. The test duration for carbon and low-alloy steels in Solution D shall be at least 17 days. For stainless steels, Ni-, Ni/Co-, Ti-, or Zr-based 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 720 hours. For testing steels at very high strength levels, at low temperatures or mild environments, it may be necessary to test for at least 21 days or longer. The duration of the test shall be mutually agreed upon among all interested parties and shall be reported with the results.

11.5.9 After the exposure, remove corrosion products from the DCB test specimen by vapor honing, by bead- or sand-blasting lightly, or by any other means that do 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. The lift-off load should be taken directly from the curve instead of attempting to correct for small (<5%) compliance error that is introduced during the measuring process.[21] Lift-off load shall be determined within three days after exposure.

11.5.10.1 The test machine load shall be calibrated per ASTM E4 (Standard Practices for Force Verification of Testing Machines).[22] Prior to applying a load, the testing machine and graph shall be set up in such a manner that zero force indication signifies a state of zero force on the specimen.

11.5.10.2 The displacement rate shall not exceed 0.5 mm/min (0.02 in/min).

11.5.10.3 When determining the equilibrium wedge-load (P), the resolution of the load on the graph or digital display shall allow load determination within ±1.0 % of the final wedge-load (P).

11.5.11 After wedge removal, open the DCB test specimen mechanically to expose the crack surfaces. This procedure may require the use of a tensile machine. For ferritic steels, this process can be made easier by chilling the DCB 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 shall show at least 2.5 mm (0.10 in) crack growth beyond the base of the chevron, EDM notch, or fatigue precrack;

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

(c) If the fracture is nonplanar at any location along its length, the raised surface shall not project beyond the "V" portion of the side groove; however, a deviation at a small point shall not invalidate the test.

(d) The crack front shall not be pinned; causes of pinning include internal cracks and fissures;

(e) The crack front should be free of significant disturbance created when the main crack dips below, or rises above, a previously formed (darker) triangular edge crack ("shark's tooth").[23] The combined width of any edge crack(s) at the crack front on the fracture surface shall not exceed 25% of the web thickness B_n for a valid test. Edge cracks lying away from the crack front (that formed earlier in the test) shall be given one-fourth the weight of those at the crack front. Thus, a DCB specimen whose earlier-formed edge cracks have a combined width exceeding 100% of the web thickness shall be invalid. For DCB test specimens with both earlier-formed edge cracks and edge cracks at the crack front, the sum of the combined widths of the earlier-formed edge cracks divided by four, plus the widths of those at the crack front, shall not exceed 25% of the web thickness, B_n, for the test to be considered valid. See Figure 12 for an example of an invalid test caused by excessive edge cracking.

NOTE: Edge cracks may not affect results in the mechanical assurance curve (see Appendix D [Nonmandatory]).

(f) The crack should not branch into one or both of the sidearms (side crack);

(g) Dry cracks are lighter colored SSC cracks that are characterized by the same topology, and extend beyond, the black SSC crack (see Figure 13). The crack length used to calculate the K_{ISSC} shall include any dry cracking.

(h) The crack front of full-thickness (curved) DCB 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 characteristics in Paragraph 11.6.1, the test shall be considered valid. The distance from the slotted end of the

DCB test specimen to the mean position of the crack front shall be measured under magnification using a dial caliper, digital caliper or comparable instrument. It is recommended, along the crack front, to use five equally spaced points across the width starting and ending at each edge and then use the average of the three middle points with the average of the two edges as the mean position of the crack front. 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 DCB test specimens shall be calculated using Equation (8):

$$K_{ISSC} \text{ (flat DCB test specimen)} = \frac{Pa(2\sqrt{3} + 2.38h/a)(B/B_n)^{1/\sqrt{3}}}{Bh^{3/2}} \qquad (8)$$

where:

K_{ISSC} = threshold stress intensity factor for SSC;
P = measured lift-off load;
a = crack length, as described in Paragraph 11.6.2;
h = height of each arm;
B = DCB test specimen thickness; and
B_n = web thickness.

Calculation example:

Data:	B = 9.53 mm	(0.375 in);
	B_n = 5.72 mm	(0.225 in);
	h = 12.7 mm	(0.500 in);
	P = 1,870 N	(421 lb_f);
	a = 46.48 mm	(1.830 in); and
Result:		
	K_{ISSC} = 35.3 MPa√m	(32.1 ksi√in).

11.6.4 The stress intensity factor for SSC of full-wall thickness (curved) DCB test specimens shall be computed using Equation (9):[24]

$$K_{ISSC} \text{ (curved DCB test specimen)} = \left(\frac{3I}{Bh^3}\right)^{1/2} K_{ISSC} \text{ (flat DCB test specimen)} \qquad (9)$$

where:

K_{ISSC} (flat DCB test specimen) is calculated using Equation (8); and

Quantity I is computed from Equation (10) as follows:

$$\begin{aligned} I = &-\frac{h}{4}\left(\frac{D^2}{4} - h^2\right)^{3/2} + \frac{D^2h}{32}\left(\frac{D^2}{4} - h^2\right)^{1/2} + \frac{D^4}{128}\sin^{-1}\left(\frac{2h}{D}\right) + \frac{h}{4}\left[\left(\frac{D}{2} - B\right)^2 - h^2\right]^{3/2} \\ &- \frac{h}{8}\left(\frac{D}{2} - B\right)^2\left[\left(\frac{D}{2} - B\right)^2 - h^2\right]^{1/2} - \frac{1}{8}\left(\frac{D}{2} - B\right)^4 \sin^{-1}\left(\frac{2h}{D - 2B}\right) \end{aligned} \qquad (10)$$

where:

B and h have values in accordance with Paragraph 11.6.3; and
D = tubular outside diameter.

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

Calculation example:

Data: D = 193.7 mm (7.625 in); and
Other data as in Paragraph 11.6.3 calculation example.
Result:
K_{ISSC} = 35.4 MPa√m (32.2 ksi√in).

11.7 Reporting of Test Results:

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

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

11.7.3 Table 6 shows the recommended format for reporting the data. In addition to items in Table 6 (Part 2), report the following for each specimen:
11.7.3.1 Actual Dimensions: height (h), specimen thickness (B), web thickness (B_n), crack length (a) and actual arm displacement (δ);
11.7.3.2 Lift-off load (P);
11.7.3.3 Start and end of test H_2S concentrations (when specified);
11.7.3.4 Three HRC hardness indentations results (when specified);
11.7.3.5 Actual test duration.

11.8 The recommended quality assurance method of evaluating the final mechanical state and validity within the test parameters of each DCB test specimen is in Appendix D.

11.9 A recommended method for determining K_{LIMIT} and $K_{Iapplied}$ is provided in Appendix E (Nonmandatory). K_{LIMIT} represents the crack initiation toughness for a specific material tested in a specific environment and estimates the minimum K_{ISSC}. $K_{Iapplied}$ is the applied stress intensity factor obtained after wedge insertion and before the environmental exposure.

Figure 12: Example of Invalid Test due to Edge Cracks Using Low-Angle Fiber Optics Illumination (See Paragraph 11.6.1[e])

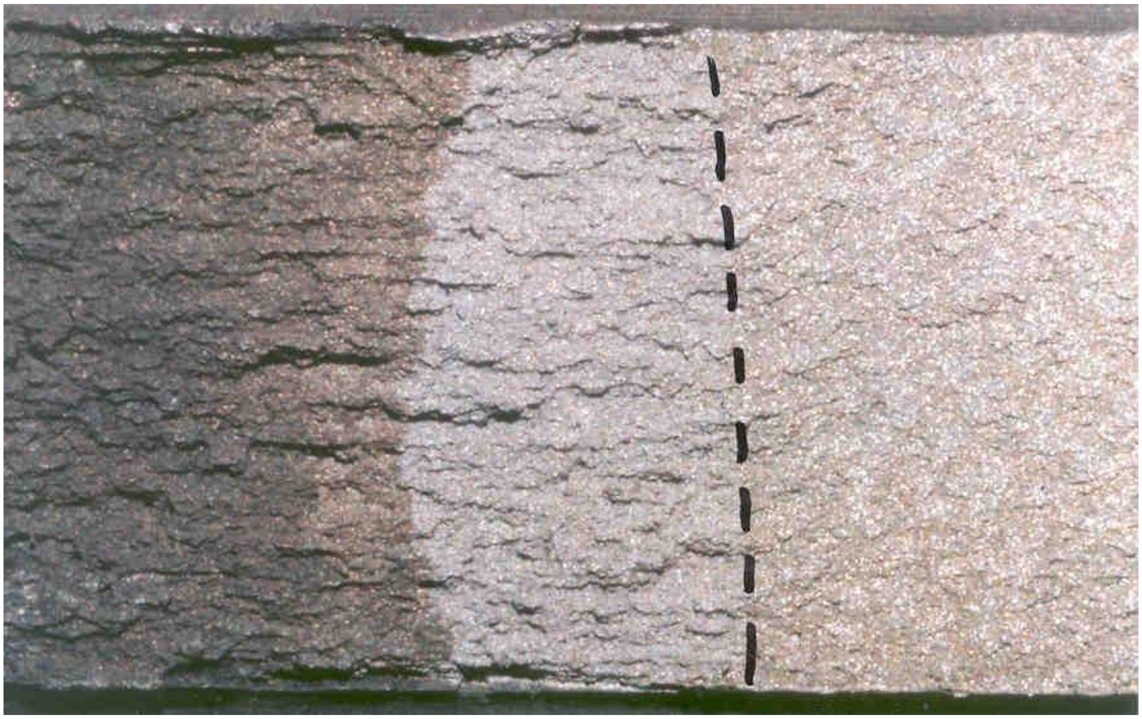


Figure 13: Example of Dry Cracking (See Paragraph 11.6.1[g])

Table 7
NACE Uniform Material Testing Report Form (Part 1): Testing in Accordance with NACE Standard TM0177[(A)] Method D—NACE Standard DCB 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.

(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 *7* (continued)
NACE Uniform Material Testing Report Form (Part 2): Testing in Accordance with NACE Standard TM0177 Method D—NACE Standard DCB Test

Lab Data for Material: ____Tested in accordance with NACE Standard TM0177(A)

DCB Specimen Geometry: ☐ Standard ☐ Nonstandard: Thickness ___________ Height (2h)_________Length_________
☐ Galvanic Couple _____________________ ☐ Fatigue Precrack ☐ EDM Crack Starter
Elastic Modulus________________________
Chemistry: ☐ Test Solution A ☐ Test Solution B ☐ Test Solution C (define) ___☐ Test Solution D_____☐ Other Test Solution ________________
☐ Outlet Trap to Exclude Oxygen ☐ Temperature Maintained 24° ± 1.7 °C (75° ± 3.0 °F)_____☐ Temperature Maintained ±1.7 °C (±3.0 °F)

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

(A) Test method must be fully described if not in accordance with NACE Standard TM0177.

(B) Location of test specimen 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 DCB test specimen at highest stress if summarizing data.

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Appendix A
Safety Considerations in Handling H_2S Toxicity
(Nonmandatory)

This appendix is considered nonmandatory, although it may contain mandatory language. It is intended only to provide supplementary information or guidance. The user of this standard is not required to follow, but may choose to follow, any or all of the provisions herein.

H_2S is perhaps responsible for more industrial poisoning accidents than is any other single chemical. A number of these accidents have been fatal. H_2S must be handled with caution and any experiments using it must be planned carefully. The OSHA maximum allowable concentration of H_2S in the air for an eight-hour work day is 20 parts per million (ppm), well above the level detectable by smell.[25] However, the olfactory nerves can become deadened to the odor after exposure of 2 to 15 min, depending on concentration, so that odor is not a completely 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 symptoms of poisoning in this range of concentration. Pulmonary complications, such as pneumonia, are strong possibilities from such subacute exposure. At 500 ppm, unconsciousness may occur in less than 15 min, and death within 30 min. At concentrations above 1,000 ppm, a single inhalation may result in instantaneous unconsciousness, complete respiratory failure, cardiac arrest, and death.

Additional information on the toxicity of H_2S can be obtained from the Chemical Safety Data Sheet SD-36[26] and from *Dangerous Properties of Industrial Materials*.[27]

Fire and Explosion Hazards

H_2S is a flammable gas and yields poisonous sulfur dioxide (SO_2) as a combustion product. In addition, its explosive limits range from 4 to 46% in air. Appropriate precautions shall be taken to prevent these hazards from developing.

Safety Procedures During Test

All tests shall be performed in a hood with adequate ventilation to exhaust all of the H_2S. The H_2S flow rates during the test 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 caustic solution needs periodic replenishing. Provision shall be made to prevent backflow of the caustic solution into the test vessel if the H_2S flow is interrupted. Suitable safety equipment shall be used when working with H_2S.

Because the downstream working pressure frequently rises as corrosion products, debris, etc., accumulate and interfere with regulation at low flow rates, particular attention should be given to the output pressure on the pressure regulators. Gas cylinders shall be securely fastened to prevent tipping and breaking of the cylinder head. Because H_2S is in liquid form in the cylinders, the high-pressure gauge must be checked frequently, because relatively little time elapses after the last liquid evaporates and the pressure drops from 1.7 MPa (250 psig) to atmospheric pressure. The cylinder shall be replaced by the time it reaches 0.5 to 0.7 MPa (75 to 100 psig) because the regulator control may become erratic. Flow shall not be allowed to stop without closing a valve or disconnecting the tubing at the test vessel because the test solution continues 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 notified so that the cylinder can be given special attention.

Appendix B
Explanatory Notes on Environmental Cracking Test Method
(Nonmandatory)

This appendix is considered nonmandatory, although it may contain mandatory language. It is intended only to provide supplementary information or guidance. The user of this standard is not required to follow, but may choose to follow, any or all of the provisions herein.

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 ensure 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 the test 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 Test Specimen

All machining operations shall 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 shall be taken. The precautions cited in this standard minimize the effects of oxygen with little increase in cost, difficulty, or complexity.

Cautionary Notes

Cleaning solvents such as 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.

Appendix C
Determination of H_2S Concentration in Test Solution by Iodometric Titration
(Nonmandatory)

This appendix is considered nonmandatory, although it may contain mandatory language. It is intended only to provide supplementary information or guidance. The user of this standard is not required to follow, but may choose to follow, any or all of the provisions herein.

This procedure details an acceptable method that may be used for the determination of the H_2S concentration in the test solution by iodometric titration.

Test Equipment

10 mL and 25mL volumetric pipettes
50 mL and 100 mL volumetric flasks
250 mL conical (Erlenmeyer) flask
100 mL beaker
25 mL burette
50 mL syringe (graduated to 60 mL)

Test Reagents

Standard 0.1 N iodine solution (0.0995 - 0.1005 N, Certified)
Standard 0.1 N sodium thiosulfate solution (0.0995 - 0.1005 N, Certified)
Standard 0.01 N iodine solution (0.0095 - 0.0105 N, Certified)
Standard 0.01 N sodium thiosulfate solution (0.0095 - 0.0105 N, Certified)
Concentrated HCl (approx. 37 wt% HCl)
Starch solution (approx. 1 wt%)

Analytical grade reagents shall be used.

Titration Procedure

C1 Table C1 gives the titration parameters required for determination of the test solution H_2S concentration, for a series of H_2S partial pressure ranges from 1.00 bar to less than 0.01 bar. The limits for each range in the series have been chosen to ensure that analytical accuracy is maintained. The range selected shall be appropriate to the test H_2S partial pressure.

Table C1
Titration Parameters for Determination of the Test Solution H_2S Concentration.

pH_2S bar	**xH_2S** mole-%	**cH_2S** mg/L	**Test Solution Sample Volume** mL	**Iodine / $Na_2S_20_3$ Concentration** N
0.40 – 1.00	40 – 100	920 – 2300	10	0.1
0.16 – 0.40	16 – 40	370 – 920	25	0.1
0.08 – 0.20	8.0 – 20	185 – 460	50	0.1
0.04 – 0.10	4.0 – 10	92 – 230	100	0.1
0.016 – 0.04	1.6 – 4.0	37 – 92	25	0.01
0.008 – 0.02	0.8 – 2.0	18 – 46	50	0.01
< 0.01	< 1.0	< 23	100	0.01

C2 Pipette 25 mL of the selected (0.1 N or 0.01 N) standard iodine solution into a conical flask.

C3 Acidify with a few drops of concentrated HCl. If sampling using a syringe (see Paragraph C4.3), weigh the conical flask containing the acidified iodine solution and record the result.

C4.1 When sampling using a volumetric pipette, transfer an initial 25-50 mL of test solution from the test vessel into a clean beaker. Rinse the beaker with the test solution and discard. Transfer a further 50-100 mL of test solution to the beaker. Fill the pipette from the beaker, rinse with the test solution and discard. Extract the required volume of test solution from the beaker via the pipette, and immediately transfer to the conical flask containing the acidified iodine solution. Record the volume of test solution transferred.

C4.2 When sampling using a volumetric flask, transfer an initial 25-50 mL of test solution from the test vessel into the flask. Rinse the flask with the test solution and discard. Transfer sufficient test solution to fill the flask to the required volume, and immediately transfer to the conical flask containing the acidified iodine solution. Record the volume of test solution transferred.

C4.3 When sampling using a syringe, extract an initial 25-50 mL of test solution from the test vessel. Rinse the syringe with the test solution and discard. Refer to the graduations on the syringe to extract the approximate volume of test solution required, and transfer directly to the pre-weighed conical flask containing the acidified iodine solution. Re-weigh the conical flask and record the result. Determine the exact volume of test solution transferred.

NOTE: Care should be taken to ensure that dissolved gases do not break out of the test solution when extracting samples via syringe.

C5 Titrate with the selected (0.1 N or 0.01 N) standard sodium thiosulfate solution until the solution changes color from dark yellow/tan to pale yellow.

C6 Add a few drops of starch solution.

NOTE: The starch addition shall be made near the end point of the titration when most of the iodine has been removed, and the color of the solution starts to fade. This is necessary due to the insolubility of the blue starch-iodine complex which may otherwise prevent some of the iodine from reacting.

C7 Continue to titrate slowly with the selected (0.1 N or 0.01 N) standard sodium thiosulfate solution until the dark blue color disappears, and the end point is reached. The end point is a milky yellow suspension of colloidal sulfur. Record the total volume of standard sodium thiosulfate solution used.

C8 Calculate the H_2S concentration (mg/L) using Equation (C1):

$$\text{H}_2\text{S concentration (mg/L)} = \frac{(\text{A} - \text{B})}{\text{C}} \text{ x } 17{,}040 \qquad \text{(C1)}$$

where:

A = Normality of standard iodine solution (equivalents/L) times the volume used (L)
B = Normality of standard sodium thiosulfate solution (equivalents/L) times the volume used (L)
C = Volume of test solution sample (L)

$$17{,}040 = \frac{34.08 \text{ (g/mole H}_2\text{S) x } 1000 \text{ (mg/g)}}{2 \text{ (equivalents/mole H}_2\text{S)}}$$

Appendix D
Recommendations for Determining Mechanical Quality Assurance of Test Results for Method D (DCB Test)[28]
(Nonmandatory)

This appendix is considered nonmandatory, although it may contain mandatory language. It is intended only to provide supplementary information or guidance. The user of this standard is not required to follow, but may choose to follow, any or all of the provisions herein.

The purpose of this informative appendix is to provide a recommended quality assurance method of evaluating the final mechanical state of the NACE TM0177 Method D (DCB) test specimen. This allows the interpreter of the DCB test results to evaluate whether the mechanical state (specimen dimensions, loading, etc.) of the DCB specimen were within the parameters of the test method after environmental exposure.

Method Procedure

The following information is required for each DCB test specimen:

Per material/grade of material (for suite of tests):

δ_{min} = minimum allowable arm displacement minus 0.0254 mm (0.001 in);
δ_{max} = maximum allowable arm displacement plus 0.0254 mm (0.001 in); and
E = measured elastic modulus or default of 2.07 x 10^5 MPa (30,000 ksi) for carbon and low alloy steels.

NOTE: Minimum and maximum allowable arm displacement shall be applied for casing and tubing material. For heavy wall coupling stock, such limits may not be applicable because of the scatter in the DCB test results. Therefore, more work is required to validate the arm displacement limits, and it should be agreed between manufacturer and customer how to proceed with the limits for mechanical compliance analysis.

Per each DCB test specimen:

P = measured lift-off load;
a_f = measured final crack length;
B = specimen thickness; and
h = arm height (½ specimen height).

Graphing

On a graph of a_f/h versus P/B, plot curves of constant δ_{max} and constant δ_{min} using Equation (D1).

$$P_i/B = \delta_m E/\left(-26.232 + 51.866\, a_i/h + 8.523\, (a_i/h)^2 + 8.5178\, (a_i/h)^3\right) \quad \text{(D1)}$$

where:

δ_m is δ_{max} or δ_{min} and
(P_i/B, a_i/h) are points on the curve of δ_{max} or δ_{min}

Each DCB test result should be recorded as a point of (P/B, a_f/h) on the graph. Equation (D1) is valid for both Chevron and EDM notch.

Evaluation

If the DCB test specimen crack surface is valid, then test results plotted between the δ_{max} and δ_{min} curves are considered mechanically valid results. This indicates the physical dimensions of the DCB specimen are correct and the required arm displacement was correctly applied by wedge insertion.

Figure D1 gives a generic example of a mechanical quality assurance graph. If cracking is valid, test results between the curves indicate the DCB test was conducted within the test parameters chosen. Test results outside the curves may have numerous causes (see Paragraph 11.6.1), including:

- DCB dimensions
- Machining
- Improper wedge placement
- Wrong arm displacement
- Invalid crack plane
- Lift-off load or final crack length improperly measured
- Wrong modulus used

Plotting a Minimum K_{ISSC} Criterion

If a minimum K_{ISSC} criterion is being applied to the test data, then that criterion can be plotted on the quality assurance graph using Equation (D2):

$$P/B = K_{ISSC}\, h^{1/2}/([2\sqrt{3}\; a/h + 2.38]\; [B/B_n]^{1/\sqrt{3}}) \qquad \text{(D2)}$$

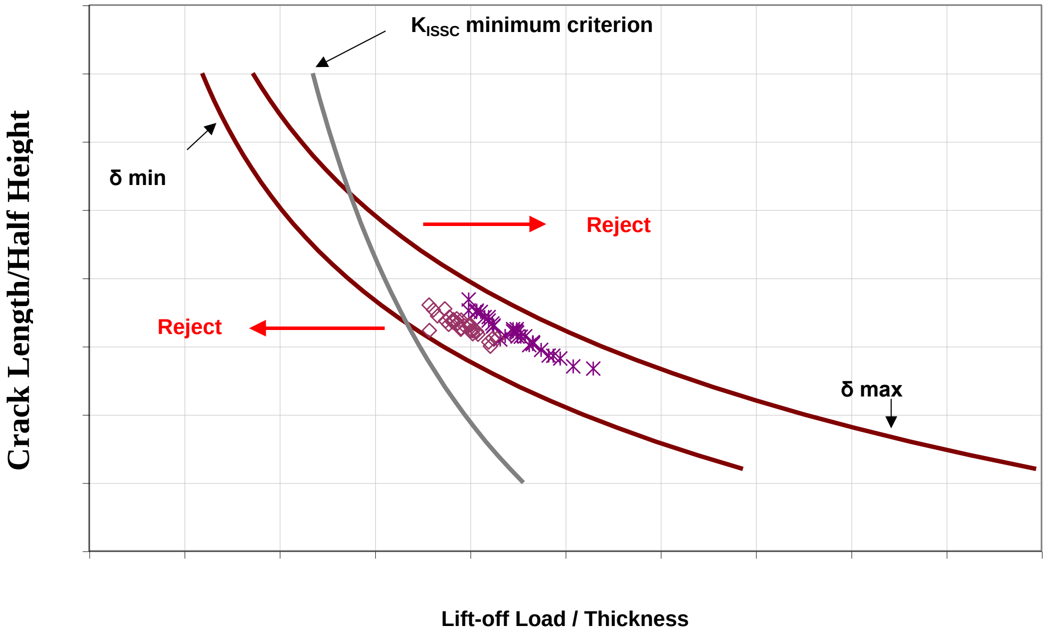

Figure D1: Example of Quality Assurance Plot

Appendix E
Recommended Method for Determining $K_{Iapplied}$ and K_{LIMIT} for the Method D (DCB) Test[29] (Nonmandatory)

This appendix is considered nonmandatory, although it may contain mandatory language. It is intended only to provide supplementary information or guidance. The user of this standard is not required to follow, but may choose to follow, any or all of the provisions herein.

The purpose of this appendix is to provide a recommended method for determining $K_{Iapplied}$ and K_{LIMIT} for the NACE Method D (DCB) test.

Definition

K_{LIMIT} is an estimate of the minimum K_{ISSC}. Therefore, K_{LIMIT} represents the crack initiation toughness for a specific material tested in a specific environment. The K_{LIMIT} is observed with decreasing arm displacement when K_{ISSC} equals $K_{Iapplied}$. The use of K_{LIMIT} as a parameter for design is under study.

$K_{Iapplied}$ is the applied stress intensity factor obtained after wedge insertion and before the environmental exposure.

Determination of K_{LIMIT}:

Optional Selection of Arm Displacement:

For the specific material and environment of exposure, K_{ISSC} results from 3 different arm displacements are required. It is recommended that valid tests at each arm displacement be conducted in triplicate. The middle arm displacement should be that specified in Section 11. The minimum difference between two arm displacements shall be at least 0.15 mm (0.006 in). Note that too small arm displacement may give no result.

Required Information:

For each DCB specimen, the following is necessary:

K_{ISSC} as obtained in accordance with Section 11.6; and
$K_{Iapplied}$ as defined in the following paragraph.

Determination of $K_{Iapplied}$:

If the fracture surface is satisfactory with respect to all the characteristics in Paragraph 11.6.1, the test shall be considered valid. The distance from the slotted end of the DCB test specimen to the mean position of the crack starter or fatigue precrack front should be measured using a dial caliper or other suitable measuring device; this measurement minus 6.35 mm (0.25 in) is the initial crack length (a_i). Use the Chevron effective starting crack of 30.02 mm (1.182 in) for any $K_{Iapplied}$ or K_{LIMIT} calculations for the Chevron DCB test specimen.

The following procedure shall be used to determine $K_{Iapplied}$ based on final valid K_{ISSC} results for greater consistency.

The final arm displacement, δ_f, shall be calculated from the lift-off load and final crack length by following Equation (E1) for flat DCB test specimens:

$$\delta_f = P\,(-\,26.232 + 51.866\ a_f/h + 8.523\ (a_f/h)^2 + 8.5178\ (a_f/h)^3)/EB \qquad \text{(E1)}$$

The calculated initial load, P_i, shall be calculated by the using Equation (E2) for flat DCB test specimens:

$$P_i = \delta_f\ EB/\ (-\,26.232 + 51.866\ a_i/h + 8.523\ (a_i/h)^2 + 8.5178\ (a_i/h)^3) \qquad \text{(E2)}$$

The $K_{Iapplied}$ of flat DCB test specimens shall be calculated using Equation (E3):

$$K_{Iapplied}\ \text{(flat DCB test specimen)} = \frac{P_i\ a_i\ (2\sqrt{3} + 2.38\ h/a_i)(B/B_n)^{1/\sqrt{3}}}{Bh^{3/2}} \qquad \text{(E3)}$$

where:

B, B_n and h have values in accordance with Paragraph 11.6.3;
P = measured lift-off load;
a_f = measured final crack length;
δ_f = calculated final arm displacement;
P_i = calculated initial load;
E = measured elastic modulus or default of 2.07×10^5 MPa (30,000 ksi) for carbon and low alloy steels; and
a_i = starting (initial) crack length.

On a graph of K_{ISSC} versus $K_{Iapplied}$, plot the line of $K_{ISSC} = K_{Iapplied}$. Then plot the K_{ISSC} versus $K_{Iapplied}$ result for each individual DCB test. Perform a linear least squares analysis on the DCB test data and determine the best fit linear equation (line). Extrapolate the best fit line to intersect with the $K_{ISSC} = K_{Iapplied}$ line. The intersection is defined as K_{LIMIT}. See Figure E1 for an example graph. The statistics of K_{LIMIT} are the same as the statistics for the best fit line.

The best fit line is given by Equation (E4):

$$K_{ISSC} = m\ K_{Iapplied} + c \qquad \text{(E4)}$$

where:

m = slope of line; and
c = intercept of the line.

The K_{LIMIT} is calculated by using Equation (E5):

$$K_{LIMIT} = c\ /\ (1 - m) \qquad \text{(E5)}$$

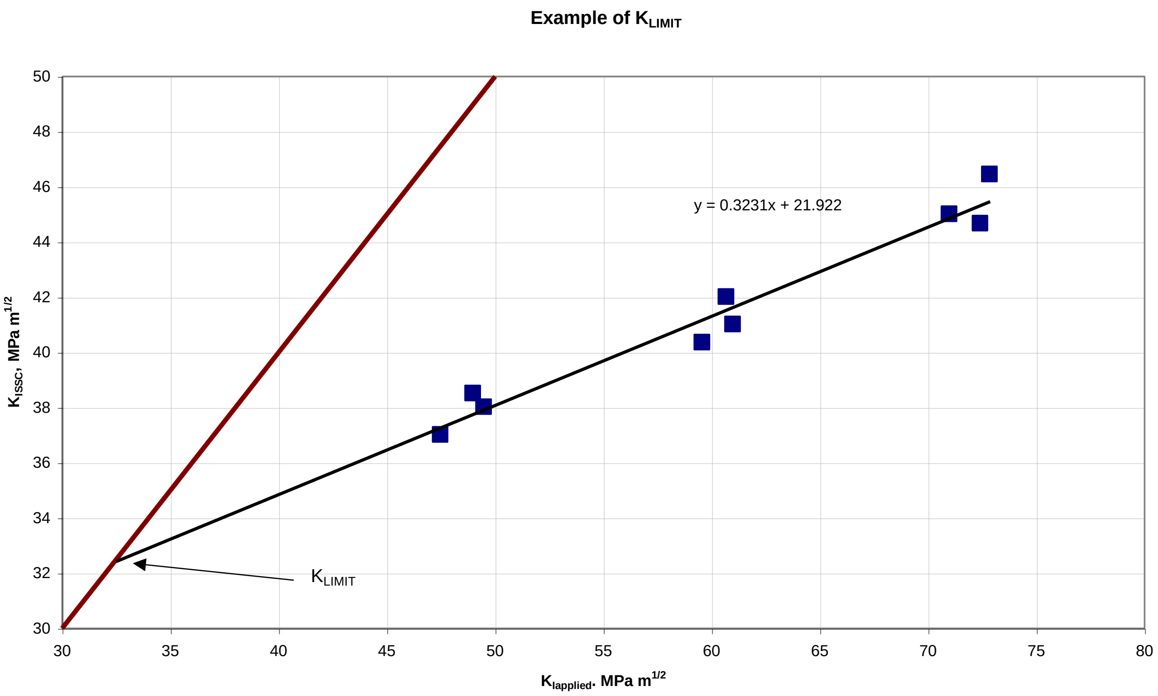

Figure E1: Example of K_{LIMIT} Determination

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