

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

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Document History:

2024-09-10: Revised by AMPP Standards Committee (SC) 08, "Metallic Material Selection & Testing"

2016-03-29: Revised by NACE Task Group (TG) 085, "Sulfide Corrosion Cracking: Metallic Materials Testing Techniques"

2005-12-03: Revised by NACE TG 085

1996: Revised by NACE TG T-1F-9, a component of Unit Committee T-1F, "Metallurgy of Oilfield Equipment"

1990: Revised by NACE TG T-1F-9

1986: Revised by NACE TG T-1F-9

1977: Developed by NACE TG T-1F-9

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Foreword

This standard addresses the testing of metals for resistance to cracking 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 temperatures near or below ambient and stress corrosion cracking (SCC) when operating at higher temperatures. In recognition of the variation in temperature and with different materials, this phenomenon is herein called environmentally assisted cracking (EAC). For the purposes of this standard, EAC includes only SSC, SCC, and hydrogen stress cracking (HSC).

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 and SCC; other failure modes (e.g., hydrogen blistering, hydrogen-induced cracking [HIC], stress-oriented hydrogen-induced cracking [SOHIC], chloride SCC, pitting corrosion, and mass-loss corrosion) are not addressed. These tests are critical assessment tools for service in H_2S environments as referenced by international standards on materials for use in H_2S -containing environments in oil and gas production.^{1,2}

The need for better understanding of the variables involved in EAC 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 EAC 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.

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 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 SCC. Such failures have historically been termed SSC even though their cause may not be hydrogen.

Scope

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 -containing environments. This standard contains methods for testing metals using uniaxial tensile, three-point bent-beam, C-ring, and double-cantilever-beam (DCB) test specimens. A separate standard, NACE TM0316,³ contains a method for testing metals using four-point bend test specimens.

This test method may be used for many purposes, and the applications of the test results are beyond the scope of this standard. Those who use the test method should be aware that, in some cases, the test results can be influenced by variations in properties among different locations in a single length of pipe, individual plate, or weld, as well as by variations within a heat of steel. When the test method is used as a basis for purchasing, the number and location of the test specimens must be carefully considered. This standard is intended for end users, manufacturers, fabricators, and testing laboratories.

Rationale

The new revision of this standard reflects industry needs and the better harmonization of testing practices. The main changes are as follows. In [Paragraph 4.5](#), a cleaning procedure was added for each test method. In [Section 6](#), the general information was clarified, and the specific test solution definition was revised with accommodation for expanded industry needs, including deaeration and saturation procedures. In [Section 7](#), clarification and standardization of testing under non-ambient conditions were added. In [Section 11](#), a detailed pre-cracking procedure was added and fracture surface evaluation exemplars were added for easier interpretation. [Appendixes A, B, and C](#) (all nonmandatory) are unchanged. However, reporting requirements for all test Methods (A, B, C, and D) were revised and qualification reporting tables were transferred to a new [Appendix D](#) (nonmandatory) titled “Recommended Reporting Template for ANSI/NACE MR0175-2021/ISO 15156:2020 Material Qualification” to enhance readability of the document. [Appendix E](#) (nonmandatory) was added, titled “Recommendations for Precracking of DCB Specimens for Method D (DCB Test).” The former Appendix E is now [Appendix F](#) (nonmandatory). Finally, [Appendix G](#) (formerly Appendix D) was revised from a nonmandatory appendix to mandatory and a second compliance method was added.

In AMPP standards, the terms *shall* and *must* are used to state requirements and are considered mandatory. The term *should* is used to state something that is recommended, but is not considered mandatory. The term *may* is used to state something considered optional.

Section 1: General

1.1 This standard covers the testing of metals subjected to tensile stresses for resistance to cracking in low-pH aqueous environments containing H₂S. Carbon and low-alloy steels are commonly tested for EAC resistance at ambient temperature where SSC susceptibility is typically high. For other types of alloys, the correlation of EAC 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 Three-Point 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 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 EAC at non-ambient temperatures and pressures.

1.3.1 For testing at ambient conditions, the test procedures can be summarized as follows: Stressed test specimens are immersed in acidified aqueous environments containing H₂S. A range of applied loads can be used to obtain EAC data.

1.3.2 For testing at non-ambient temperatures and pressures see [Section 7](#). All methods (A, B, C, and D) are adaptable to this technique.

1.4 This standard may be used for material release or acceptance testing to ensure that product meets a certain minimum level of EAC resistance in accordance with API⁽¹⁾ Specification 5CT,⁴ ISO⁽²⁾ 11960,⁵ or as specified by the user or purchaser. This standard may also provide a quantitative measure of the product’s EAC resistance for research or informational purposes. This rating may be based on:

⁽¹⁾ American Petroleum Institute (API), 1220 L St. NW, Washington, DC 20005-4070, www.api.org.

⁽²⁾ International Organization for Standardization (ISO), Chemin de Blandonnet 8, Case Postale 401, 1214 Vernier, Geneva, Switzerland, www.iso.org.

Method A—The highest no-cracking uniaxial tensile stress in 720 hours.

Method B—The statistically based critical stress factor (S_c) for a 50% probability of cracking in 720 hours.

Method C—The highest no-cracking circumferential stress in 720 hours.

Method D—The average K_{ISSC} threshold stress intensity factor for SSC.

NOTE: Additional exposure time can be required when testing corrosion-resistant alloys (CRAs).² Further guidance is provided in ISO 15156-3:2020 Technical Circular 2:2022.

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

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 cracking by HIC, SOHIC, and hydrogen blistering. This is especially true for lower-strength steels not usually subject to SSC. HIC and SOHIC 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).⁶

2.1.2 The test environment may corrode some alloys that normally do not corrode in actual field service and thereby induce EAC in alloys that ordinarily do not undergo EAC. 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 EAC susceptibility can be an important parameter. The fracture path in the test specimen should match that 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 EAC in some nickel-based corrosion-resistant alloys (CRAs) and reduced EAC 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.

2.2.6 Maximum no-cracking stresses for a specified exposure period should be considered apparent EAC threshold stresses. Longer exposure times or a greater number of test specimens may result in lower threshold values.

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

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

- 2.2.9** Some materials can require considerably more exposure time than others for determination of EAC resistance.

Section 3: Reagents

3.1 Reagent Purity

- 3.1.1** Sodium chloride (NaCl), acetic acid (CH₃COOH), sodium acetate (CH₃COONa), hydrochloric acid (HCl), and sodium hydroxide (NaOH) shall be reagent-grade chemicals. H₂S gas shall be chemically pure (CP grade), 99.5% minimum purity. Carbon dioxide (CO₂) and other pure component gases used for H₂S-containing gas mixtures shall be 99.998% minimum purity. (See [Appendix B](#) [Nonmandatory]).
- 3.1.2** The test water shall be distilled or deionized and of quality equal to or greater than ASTM⁽³⁾ Type IV (ASTM D1193⁷).
- 3.1.3** Inert gas (nitrogen, argon, or other suitable nonreactive gases) shall be 99.998% minimum purity.

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 have the same orientation with similar microstructure and mechanical properties.
- 4.2** When specified, tensile testing in accordance with standard test methods such as ASTM A370⁸ shall be used to determine base material properties. At least two test specimens should 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 EAC 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 EAC susceptibility. Consequently, when specified for qualification of new materials, 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 E18⁹ or ISO 6508¹⁰ (Rockwell) or ASTM E384¹¹ or ISO 6507¹² (Vickers) and reported. 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.

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 or ISO standard and reported.

⁽³⁾ASTM International (ASTM), 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959, www.astm.org.

4.5 Specimen Cleaning and Storage

- 4.5.1** Storage between machining and testing shall be in a non-corrosive environment, e.g., low-humidity air, dry nitrogen, and inhibitor-free oil. The procedure for storage should be documented.
- 4.5.2** Prior to any test specimen dimensional measurements, the specimen shall be cleaned so as to be free of corrosion products and particulates.
- 4.5.3** For test Methods A, B, C, and Method D without fatigue pre-cracking, immersion cleaning typically comprising the application of a degreasing agent and rinsing with an appropriate solvent shall be used to ensure that the test specimens are free of any contaminant, including particulates, oils, greases, or other residual surface films, prior to testing.
- 4.5.4** For Method D with fatigue pre-cracking:
 - a) Prior to fatigue pre-cracking by immersion cleaning (see Paragraph 4.5.3).
 - b) Prior to wedge insertion by surface solvent wiping only.
 - c) Prior to immersion in the test solution by surface solvent wiping only.
- 4.5.5** The cleanliness shall be verified using an appropriate method such as ASTM F21.¹³
- 4.5.6** After cleaning, specimen handling shall be in a manner that prevents contamination.

NOTE: Powder from powdered gloves can contaminate the specimen surface.

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** The test vessel should be configured to ensure a homogeneous test solution. In large test vessels, this may be achieved by continuous stirring of the test solution throughout the test.
- 5.3** Test vessels shall be capable of being purged to remove oxygen before beginning the test and of keeping air out during the test. (See [Appendix B](#) section titled “Reasons for Exclusion of Oxygen”).
- 5.4** Test vessels shall be sized to maintain the minimum test solution volume relative to the test specimen surface area to standardize the drift of pH with time. (See each test method for minimum requirements).
- 5.5** All test equipment shall be resistant to the test environment for the duration of the test. New polymethylmethacrylate (also known as acrylic) vessels shall be pre-treated with a 50% acetic acid solution for a minimum of 1 hour.
- 5.6** 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.
- 5.7** Rigid electrical insulating materials not exhibiting relaxation or flow under load should be selected for loading or deflecting the test specimen.
- 5.8** Galvanic Coupling Testing for EAC Resistance
 - 5.8.1** A galvanic couple between dissimilar metals may result in a change in the susceptibility to EAC (see [Paragraph 2.2.2](#)).
 - 5.8.1.1** To evaluate this, one or more iron or steel coupons having a total surface area between 0.5 and 1 times the exposed area of the test specimen shall be attached in direct electrical contact with the test specimen.

- 5.9 All measuring equipment shall be calibrated or verified.
- 5.10 Vessels and solution-wetting accessories shall be clean and free of contaminants, e.g., particulates, inhibitor residuals, and oils.

Section 6: Test Solutions

6.1 General

- 6.1.1 All reagents added to the test solutions shall be measured to $\pm 1.0\%$ of the quantities specified for the specific test solution.
- 6.1.2 For testing under ambient conditions, the test solution temperature 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. For testing under non-ambient conditions, the test temperature range shall be determined in accordance with Section 7. Test solution temperature shall be monitored periodically throughout the test duration and reported.
- 6.1.3 The test solution and test gas composition used shall be reported.
- 6.1.4 Pre-mixed commercial test gas mixtures shall have a composition certified by analysis. Continuously blended test gas mixtures shall have a composition verified by measurement. Each pure component gas used for continuously blended test gas mixtures shall be certified to meet minimum reagent purity requirements.
- 6.1.5 The dissolved H₂S concentration in the test solution shall be measured by iodometric titration or by other suitable methods. Acceptable iodometric titration and reduced concentration of H₂S procedures are detailed in [Appendix C](#) (Nonmandatory).
- 6.1.6 The test solution pH shall be measured and reported as specified for each test solution. The pH measurement shall be in accordance with an international or national standard (e.g., ASTM D1293¹⁴ or ISO 10523¹⁵). Commercially available pre-prepared buffer solutions are acceptable.
- 6.1.7 Saturation with the H₂S-containing test gas shall be either verified or validated in accordance with [Paragraph 6.8](#).
- 6.1.8 The start of the test shall be defined as the time that saturation is achieved.

6.2 Test Solution A

- 6.2.1 Test Solution A is an acidified aqueous brine solution with an H₂S partial pressure specified by the user or purchaser.
- 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 CH₃COOH dissolved in 945 g of distilled or deionized water).
- 6.2.3 The initial test solution pH (before transfer to the test vessel) shall be between 2.6 and 2.8 and shall be reported. Alteration of test solution composition to adjust the pH is not permitted. If the measured solution pH is out-of-range, then the solution shall be discarded. During the test, the pH may increase but the final pH (at the end of the test) shall not exceed 4.0 and shall be reported. If the pH exceeds 4.0, the test is invalid. If the test-solution-volume-to-test-specimen-surface-area ratio is maintained and the dissolved oxygen requirements are met, then the pH will not exceed this value.

NOTE: Historically, Test Solution A has been specified with chemically pure H₂S.

6.2.4 The test gas shall consist of one of the three following options as specified by the user or purchaser. After saturation, the gas should be bubbled continuously at a rate that maintains saturation (see [Paragraph 6.8](#)).

6.2.4.1 Chemically pure H₂S.

6.2.4.2 A mixture of H₂S and nitrogen, with H₂S content sufficient to produce the specified H₂S partial pressure.

6.2.4.3 A mixture of H₂S and CO₂, with H₂S content sufficient to produce the specified H₂S partial pressure.

6.3 Test Solution B

6.3.1 Test Solution B is an acidified and buffered aqueous brine solution with an H₂S partial pressure specified by the user or purchaser.

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 CH₃COOH, and 4.1 g of CH₃COONa dissolved in 921 g of distilled or deionized water).

6.3.3 The initial test solution pH (before transfer to the test vessel) shall be between 3.4 and 3.6 and shall be reported. Alteration of test solution composition to adjust the pH is not permitted. If the measured solution pH is out-of-range, then the solution shall be discarded. During the test, the pH may increase but the final pH (at the end of the test) shall not exceed 4.0 and shall be reported. If the pH exceeds 4.0, the test is invalid. If the test-solution-volume-to-test-specimen-surface-area ratio is maintained and the dissolved oxygen requirements are met, then the pH will not exceed this value.

NOTE: Historically, Test Solution B has been specified with chemically pure H₂S.

6.3.4 The test gas shall consist of one of the three following options as specified by the user or purchaser. After saturation, the gas should be bubbled continuously at a rate that maintains saturation (see Paragraph 6.8).

6.3.4.1 Chemically pure H₂S.

6.3.4.2 A mixture of H₂S and nitrogen, with H₂S content sufficient to produce the specified H₂S partial pressure.

6.3.4.3 A mixture of H₂S and CO₂, with H₂S content sufficient to produce the specified H₂S partial pressure.

6.4 Test Solution C

6.4.1 Test Solution C is an aqueous brine solution with a chloride content, H₂S partial pressure, and pH specified by the user or purchaser.

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

6.4.3 The initial solution pH (before transfer to the test vessel) shall be adjusted by addition of HCl or NaOH to achieve the specified initial pH value ± 0.2 pH units and shall be reported. Test solution

pH shall be measured at the start of the test and at the end of the test (final pH) and shall be reported. Periodic pH measurements shall be performed and, when necessary, further additions of HCl or NaOH made to re-adjust the pH. All measured test solution pH values and any pH adjustments made during the test shall be documented.

- 6.4.4** Test gas shall consist of a mixture of H₂S and CO₂, with H₂S content sufficient to produce the specified H₂S partial pressure. 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 (see Paragraph 6.8).

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 in 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 an aqueous brine solution with an H₂S partial pressure and pH specified by the user or purchaser.
- 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 CH₃COONa dissolved in 946 g of distilled or deionized water).
- 6.5.3** The initial solution pH (before transfer to the test vessel) shall be adjusted by addition of HCl or NaOH to conform to the specified initial pH requirements and shall be reported. The test solution pH shall be measured at the start of the test and at the end of the test (final pH) and shall be reported. When adjustment of the test solution pH during the test is specified, periodic pH measurements shall be performed and, when necessary, further additions of HCl or NaOH made to re-adjust the pH. All measured test solution pH values and any pH adjustments made during the test shall be documented.
- 6.5.4** The test gas shall consist of one of three following options as specified by the user or purchaser. After saturation, the gas should be bubbled continuously at a rate that maintains saturation (see Paragraph 6.8).
- 6.5.4.1** Chemically pure H₂S.
- 6.5.4.2** A mixture of H₂S and nitrogen, with H₂S content sufficient to produce the specified H₂S partial pressure.
- 6.5.4.3** A mixture of H₂S and CO₂, with H₂S content sufficient to produce the specified H₂S partial pressure.

NOTE 1: Historically, Test Solution D has been used for verification testing of API 5CT Grade C110 using a test gas consisting of 7.0 ± 0.3% H₂S with balance of N₂ gas, an initial solution pH 4.0, and a final pH requirement below 4.6.

NOTE 2: Test Solution D is specified for SSC Region 1 and 2 qualification testing of carbon and low-alloy steels in accordance with ANSI/NACE MR0175-2021/ISO 15156:2020 Part 2, Clause B.3. and Table B.1.

- 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 achieve dissolved oxygen levels in the test solution (immediately prior to the introduction of the test gas) of less than 50 ppb for low-alloy steels up to 552 MPa (80 ksi) specified minimum yield strength (SMYS) and 10 ppb for low-alloy steels above 552 MPa (80 ksi) SMYS and corrosion resistant alloys.

Each laboratory shall have documented, equipment-specific procedures to achieve the required dissolved oxygen levels. Measurement of dissolved oxygen levels shall conform to one of the following practices.

6.7.1.1 Dissolved oxygen analytical equipment, provided that it has a lower detection limit less than or equal to 4 ppb and an error less than or equal to 4 ppb in the range of 0 – 50 ppb dissolved oxygen.

6.7.1.2 Color-indicating ampoules and appropriate color comparators, provided that the apparatus and methodology comply with ASTM D5543¹⁶ or comparable international standard.

6.7.2 Deaeration Procedures

6.7.2.1 The test solution shall be previously deaerated in a sealed vessel that is sparged 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(s) containing the test specimen(s) only, shall be purged separately with inert gas at a rate of at least 100 mL/min per liter of test vessel volume 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; see [Paragraph 6.7.1](#).

6.7.3 Significant oxygen contamination (>> 50 ppb) is evident by a cloudy (opaque) appearance of the test solution when the H₂S gas enters the test vessel. An opaque appearance of the test solution upon H₂S entry shall invalidate the test. The test specimen(s) 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.

6.8 Solution Test Gas Saturation Requirements

6.8.1 For Solution A, B, and D, the minimum required dissolved H₂S concentration at 100 kPa H₂S partial pressure shall be 2,300 mg/L. For H₂S partial pressures below 100 kPa, the minimum required dissolved H₂S concentration shall be proportional to the H₂S partial pressure of the test gas (Equation [1]).

$$\text{Minimum dissolved H}_2\text{S (mg/L)} = 2,300/100 \times \text{pH}_2\text{S (kPa)} \quad (1)$$

NOTE: For example, the required minimum H₂S concentration in solution for 7% H₂S purge gas with a partial pressure of 7 kPa is 161 mg/L = 2,300/100 x 7.

6.8.2 For Solution C and other test solutions with a chloride content determined by the intended service

environment, the dissolved H₂S concentration at saturation can vary significantly; for these solutions, the resource for determining the expected H₂S saturation shall be reported. Resources for determining the anticipated aqueous H₂S concentration include reported data on H₂S saturation in relevant environmental conditions and thermodynamic calculations.

- 6.8.3** Saturation shall occur within an hour of H₂S gas contact with specimens in test vessels up to 1 L (see NOTE). For larger test vessels, saturation can require more than two hours.

NOTE: Minimum dissolved H₂S concentration for Method A (approximately 1/3 L) can be reached by sparging at 100 mL/min for 60 minutes.

- 6.8.4** The test solution shall be sparged with the H₂S-containing test gas at a documented minimum flow rate. Saturation with the H₂S-containing test gas (see Paragraphs 6.8.1 and 6.8.2) shall be either:
- a) Verified by extracting test solution samples to determine that the dissolved H₂S concentration, at the start of the test and at the end of the test, meets the minimum required value; see [Paragraph 6.1.5](#); or
 - b) Validated by following a documented, equipment-specific test procedure that both achieves and maintains the minimum required dissolved H₂S concentration throughout the test duration. The equipment-specific test procedure shall be validated by extracting test solution samples to determine that the dissolved H₂S concentration at the start of the test, after 24 hours, weekly, and at the end of the test, meets the minimum required value.

Section 7: Testing under Non-Ambient Conditions

- 7.1** Testing under non-ambient conditions requires explicit specification from the user or purchaser and shall include the following: temperature, pressure, H₂S quantity, CO₂ quantity, other gas compositions, presence of elemental sulfur, which test method, resources for environment verification, methods for environment verification, test duration, post-test assessment requirements, and reporting requirements that exceed those listed.
- 7.2** The dominant cracking mechanisms for most classes of materials in the presence of H₂S can 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 or below ambient 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 H₂S partial pressure exceeding 100 kPa (absolute) (14.5 psia), the following modified techniques are available.

7.3 Approach

- 7.3.1** Testing at elevated temperatures and pressures involves additional safety considerations compared to ambient temperature and atmospheric pressure testing. While some general guidance is given herein, it may not address all aspects and shall be supplemented to comply with local safety requirements.
- 7.3.2** Methodology to establish the target environmental conditions varies and best practices vary significantly with the temperature and pressure of the exposure test. General approaches to establishing environments at elevated temperature and pressure are reviewed in some literature publications.^{17,18,19,20,21,22} The procedure used to model, establish, maintain, and verify the environmental conditions shall be documented.

- 7.3.2.1** If a thermodynamic calculation is performed and used to determine or guide procedural steps for establishment of the exposure environment, the calculation details and results shall be reported.

- 7.3.2.2** Procedural steps taken by the laboratory to establish and maintain (i.e., replenish) the environment shall be documented. Reporting shall include the temperature, pressure, and gas compositions used at each procedure step.
- 7.3.2.3** Verification of the environmental conditions shall be documented. Methods to verify elevated temperature and pressure sour environment H₂S presence vary and may include compositional measurements and total pressure measurements. Commonly used methods include colorimetric detection, gas chromatography, iodometric titration, and verification of pressures benchmarked to thermodynamic predictions. Gas chromatography is considered the most accurate of the methods but may have associated sampling and measurement challenges due to H₂S sequestration and water condensation. Similarly, colorimetric detection tubes for gas analysis also require cooling the gas to near ambient temperature and may have associated water condensation and H₂S sequestration issues. Aqueous H₂S concentration measurements via iodometric titration or colorimetric detection are also challenging due to sampling issues such as gas flashing during liquid removal from the pressure vessel. Verification of thermodynamic pressure targets relies on the accuracy of the original thermodynamic calculation and may be of limited use if the partial pressures of H₂S and CO₂ are small compared to pressure reading accuracy.
- 7.3.3** When assessing materials more sensitive to SSC at temperatures below the intended test temperature, H₂S should only be introduced once the test cell and fluids are at the target temperature or another selected intermediate temperature. H₂S should be removed via purging with inert gas at the termination of the test prior to lowering the temperature below the target temperature or a selected intermediate temperature.
- 7.3.4** Because H₂S may be consumed during the test, gas replenishment and continuous gas bubbling techniques are described. The H₂S 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 H₂S in the test environment. Guidance is given in [Paragraph 7.8.5](#) on measures that experience has shown to be appropriate for maintaining the required H₂S partial pressure in certain ranges of temperature and pressure, but it may not address all cases.

7.4 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.

- 7.4.1** 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.
- 7.4.2** 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.
- 7.4.3** A bursting (rupture) disc or pressure-relief valve should be used for safety reasons.
- 7.4.4** The pressure-measuring device shall have an accuracy of ±1% of the test 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.
- 7.4.5** Non-metallic Materials Use
- 7.4.5.1** Elastomeric seal materials, if used, shall be resistant to the test environment over the duration of the test.

7.4.5.2 Thermoplastic and elastomer materials can absorb H_2S and affect the H_2S content of the test environment.

7.4.6 The test vessel shall be sized to maintain a minimum test solution volume of 20 mL/cm² of test specimen surface area when testing carbon and low alloy steels. CRA materials may not require a similar solution volume-to-specimen surface area ratio, but consideration should be given to changes in the environment over the duration of the test. The use of any solution volume-to-specimens surface area ratio less than 20 mL/cm² shall be reviewed in advance with the user or purchaser and reported.

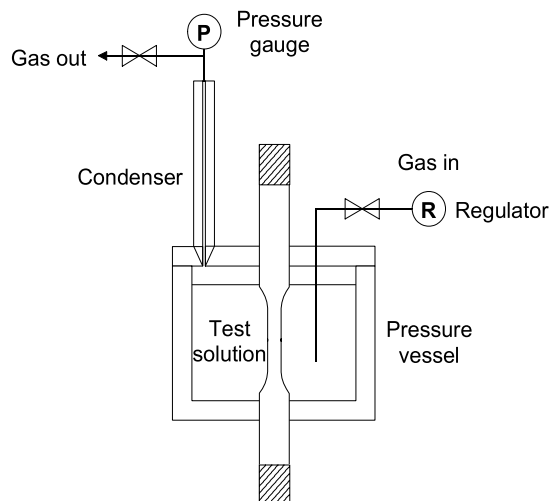


Figure 1: Schematic Arrangement of Non-Ambient Test Equipment for Method A—NACE Standard Tensile Test

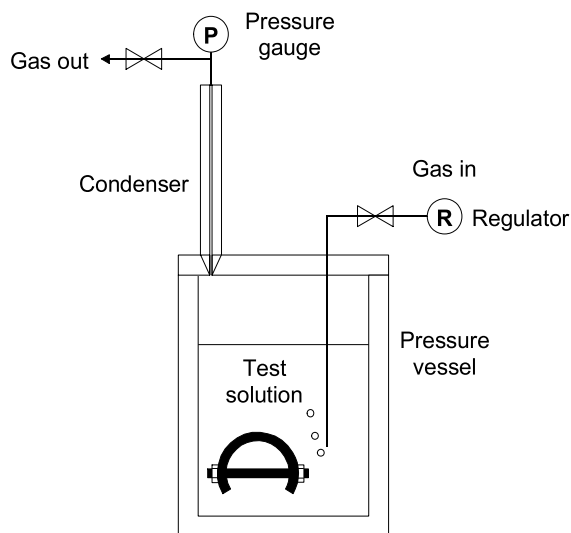


Figure 2: Schematic Arrangement of Non-Ambient Test Equipment for Method B—NACE Standard Three-Point Bent-Beam Test, Method C—NACE Standard C-Ring Test, and Method D—NACE Standard Double-Cantilever-Beam (DCB) Test

7.5 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 ambient-temperature methods. The test solution may also contain elemental sulfur.^{2,23,24,25}

NOTE: Acetate buffers may result in significant corrosion of carbon and low alloy steel and certain CRA material specimens at elevated temperatures. Further guidance on testing environments is provided in ANSI/NACE MR0175-2021/ISO 15156:2020 Part 3 Annex D.

7.5.1 The initial pH of the solution shall be measured before transfer to the test vessel. For test gas with partial pressure of CO₂, the pH shall be measured after equilibrating the solution with CO₂ gas at ambient temperature and pressure. The pH of the solution after test shall be measured in the same manner on a solution sample equilibrated with CO₂ gas at ambient temperature and pressure. The CO₂ equilibrated pH values shall be reported. Additional pH measurements taken at temperature and pressure, using commercial or proprietary devices, or using open cup measurements are permitted and may be reported as such for informational purposes.

7.5.2 Brine solutions with total dissolved solids beyond saturation at ambient temperature may be required for testing. Handling of these solutions with respect to establishing the target environment requires care and the approach shall be reported; some approaches that have been used successfully include:

7.5.2.1 Heating the solution prior to transfer to the vessel to a higher temperature, up to 60 °C (140 °F), to dissolve the salts.

7.5.2.2 Adding the quantity of salts beyond saturation directly to the vessel and transferring in the remaining solution. Agitation of the solution within the vessel after transfer and after heating of the solution is recommended to ensure dissolution of the solids.

7.5.2.3 Preparation and transfer of a diluted solution that is then concentrated by heating the solution and allowing a controlled quantity of steam to escape the vessel. If this approach is used, the water escaping the system shall be measured and reported.

7.5.3 If direct measurement of aqueous H₂S concentration is used to verify the test environment (see [Paragraph 7.3.2.3](#)), the resource for determining the expected H₂S saturation shall be reported. Resources for determining the anticipated aqueous H₂S concentration include reported data on H₂S saturation in relevant environmental conditions and thermodynamic calculations.

NOTE: Confirmation of aqueous concentration of dissolved H₂S at elevated temperature and pressure presents difficulties with regard to safety and methodology. Aqueous H₂S concentration is strongly dependent on temperature, pressure, and brine composition. For high partial pressures of H₂S, a direct measurement may not be possible, thermodynamic modeling may be needed to verify the environment.

7.6 Test Gas

The test gas is usually a mixture of two or more of the following: H₂S, CO₂, and inert gas such as N₂ or Ar. At low H₂S partial pressures, tests in inert gas without CO₂ require careful interpretation because of corrosion product solubility effects.²⁶ 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. As noted in [Paragraph 7.8.4](#), the presence of water vapor pressure at equilibrium shall be accounted for when determining the gas composition and loading methodology.

7.7 Test Stresses

The test stresses should 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. Stressing of specimens requires a demonstrated and documented procedure. Thermal expansion or creep of fixturing and specimens can vary by test method, application of stress, and specimen and fixture geometries and should be addressed in this procedure. Steps to measure and adjust for thermal expansion or creep shall be documented; if no steps are performed, this shall be noted.

7.8 Test Procedure

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

- 7.8.1 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.
- 7.8.2 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 (437 °F).
- 7.8.3 Deaeration requirements are given in [Paragraph 6.7](#).
- 7.8.4 The H₂S partial pressure, p_{H₂S}, in the test environment may be determined by thermodynamic calculation or by the following methods:

7.8.4.1 Test vessel heated before test gas admission (for continuously flowing systems).

7.8.4.1.1 The test vessel under vacuum shall be heated with valves closed to test temperature and stabilized. System pressure (the vapor pressure of the test solution), P₁, shall be measured.

7.8.4.1.2 Test gas shall be admitted to the vessel until the test pressure, P_T, is reached and continuously purged thereafter.

7.8.4.1.3 The H₂S partial pressure, p_{H₂S}, in the test environment is approximated by Equation (2):

$$p_{H_2S} = (P_T - P_1) (X_{H_2S}) \quad (2)$$

where:

P_T = total absolute test pressure;

P₁ = vapor pressure above the test solution; and

X_{H₂S} = mole fraction of H₂S in the test gas.

7.8.4.2 Calculation of H₂S in situ.

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

7.8.4.2.2 The H₂S partial pressure, p_{H₂S}, in the test environment is approximated by Equation (3):

$$p_{H_2S} = (P_T) (X_{H_2S}) \quad (3)$$

where:

P_T = total absolute test pressure (water vapor pressure may be subtracted if H_2O is not measured in analysis); and

X_{H_2S} = mole fraction of H_2S in the equilibrated test vessel gas.

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

NOTE: Batch addition of H_2S may result in partitioning to the aqueous phase.

7.8.5 Test gas shall be replenished as needed to maintain the required test conditions (primarily H_2S partial pressure) as outlined in [Paragraph 7.3](#). Testing of CRAs at H_2S partial pressures below 3.4 kPa (absolute) (0.5 psia) or carbon and alloy steels at H_2S partial pressures below 100 kPa (absolute) (14.5 psia) may warrant such replenishment. Test solution loss should be mitigated and ingress of oxygen during test gas replenishment shall be avoided.

7.8.5.1 Methods to replenish the gas that have been used successfully include continuous gas bubbling at 0.5 to 1.0 mL/min or periodic test gas replenishment once or twice weekly.

NOTE: Purging dry gas into a high chloride solution may result in blockage. Humidifying gas before introduction into the test cell has been used successfully to avoid this issue.

7.8.6 The test duration shall be as specified by the user or purchaser. The start of test shall be measured from the time that all test conditions are achieved.

7.8.7 The test temperature shall be maintained within $\pm 3^\circ C$ ($\pm 5^\circ F$) of the specified test temperature for temperatures below $90^\circ C$ ($194^\circ F$) or within $\pm 5^\circ C$ ($\pm 9^\circ F$) of the specified test temperature for temperatures at or above $90^\circ C$ ($194^\circ F$). The test temperature shall be monitored at least once daily during business hours throughout the test duration and recorded.

7.8.8 Pressure shall be monitored at least once daily during business hours throughout the test duration and recorded. If test pressure falls by more than 14 kPa (2 psia) or 2% of total test pressure, whichever is greater, below the required test pressure, the test gas should be added or replenished. As noted in [Paragraph 7.3](#), replenishment steps shall be documented.

7.8.9 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.

7.8.10 Examination of the specimens shall include inspection for cracking as detailed in each method. In addition, for CRA materials, visual observation of pits or other notable features shall be reported. An assessment of the severity of any corrosion pitting can be made by determining the maximum pit depth and evaluating the shape of the pits (see ASTM G46²⁷ / ISO 11463²⁸).

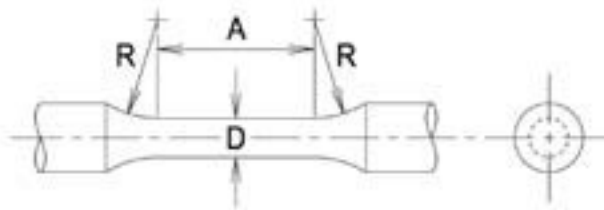
Section 8: Method A—NACE Standard Tensile Test

8.1 Method A, the NACE standard tensile test, provides for evaluating metals for EAC resistance under uniaxial tensile loading. It offers a simple unnotched test specimen with a well-defined stress state. EAC susceptibility with Method A is usually determined by time-to-fracture. Tensile test specimens loaded to a particular stress level give a cracking/no-cracking test result. When multiple test specimens are tested at varying stress levels, an apparent threshold stress for EAC can be obtained.²⁹

- 8.1.1** This section sets forth the procedure for testing at ambient temperature and atmospheric pressure. Special considerations for testing at non-ambient temperature and pressure are set forth in Section 7.

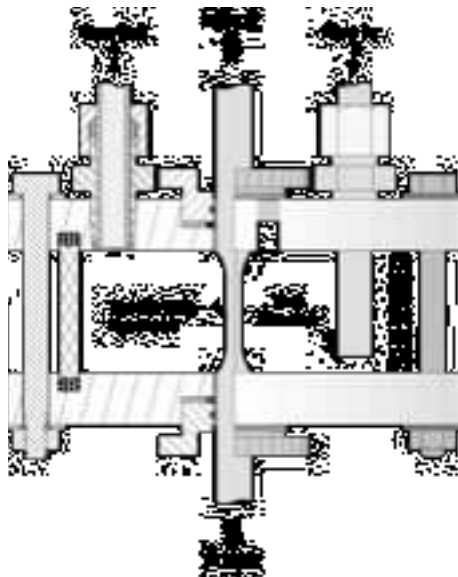
8.2 Test Specimen

- 8.2.1** The available product form of the test material often restricts selection of test specimens. The orientation of the test specimen can affect the results and shall be reported.
- 8.2.2** The reduced 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 ± 0.13 mm (1.00 ± 0.005 in) long. A subsize tensile test specimen with reduced section of 3.81 ± 0.05 mm (0.150 ± 0.002 in) in diameter by 25.4 ± 0.13 mm (1.00 ± 0.005 in) long is acceptable. Reduced section diameter and length shall be reported. After machining, store the test specimens; see [Paragraph 4.5.1](#).



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)
A	25.4 ± 0.13 mm (1.00 ± 0.005 in)	25.4 ± 0.13 mm (1.00 ± 0.005 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 reduced section (fillet radius) shall be at least 15 mm (0.60 in) to minimize stress concentrations and fillet cracking.

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

- a) Eliminate undercutting of fillet radii in machined test specimens; and
- b) Machine the test specimen reduced 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 reduced section.

- 8.2.4** The ends of the test specimen shall 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 shall be fabricated in such a way as to avoid overheating, and to minimize any incidental cold working in the reduced section.

- 8.2.6** For all materials, the average surface roughness of the reduced section shall be 0.25 μm (10 μin) or finer, as defined by the Ra value in ISO 21920-2.³⁰ 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 shall 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 times to fracture 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 or shoulders of the test specimen but shall not be used in the reduced section.

- 8.2.9** Test Specimen Cleaning – see [Paragraph 4.5](#).

- 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).³¹

- 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 loads can increase the applied test stress and may lead to specimen fracture, and rejection of a material that otherwise would be acceptable 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 increases the likelihood 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:

- 8.4.5.1.1** Before the first calibration and before any calibration following modifications or maintenance operations impacting the elastic compliance of the ring, proof rings shall be preconditioned by stressing at least 10 times to 110% of the maximum calibration load 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 outside 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 outside diameter, the calibration deflection, calibration load, and test load shall be specified.
- 8.4.5.1.3** Proof rings shall be uniquely identified, and their calibration data shall be traceable. For each test performed, the proof ring used in that test shall be traceable.
- 8.4.5.1.4** The proof ring deflection should be measured during the test or at the test completion. 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.

- 8.4.5.2** The deflection should be monitored when the applied stress exceeds 90% of the actual yield strength.
- 8.4.6** The test specimen shall be electrically isolated from dissimilar metals in contact with the test solution.
- 8.4.6.1** The seals around the test specimen shall 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 dissimilar metal, it shall be electrically isolated from the test specimen. The stressing fixture may be coated with a nonconductive impermeable coating.
- 8.4.7** The test vessel shall be sized to maintain a minimum test solution volume of 20 mL/cm² of test specimen surface area.

8.5 Stress Calculations

Loads for stressing tensile test specimens shall be determined from Equation (4):

$$P = S \times A \quad (4)$$

where:

P = load;

S = applied stress; and

A = actual cross-sectional area of the reduced section.

8.6 Testing Sequence

- 8.6.1** The tensile test specimen shall be cleaned (see Paragraph 4.5) and the minimum reduced section diameter of the specimen shall be measured. The specimen load shall be calculated (Equation [4]) for the desired stress level.
- 8.6.2** The test specimen shall be 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 by more than 2%, the test shall be run at the new load or discarded.
- 8.6.5** Deaeration and solution transfer requirements are given in [Paragraph 6.7](#).
- 8.6.6** The test solution shall then be saturated with H₂S per the requirements in [Paragraph 6.8](#).
- 8.6.7** The timing for the start of the test is defined in [Paragraph 6.1.8](#).
- 8.6.8** The termination of the test shall be at tensile test specimen fracture or after 720 hours, whichever occurs first.
- 8.6.9** When needed, additional tensile test specimens shall be tested to closely define the threshold stress for EAC.

8.7 Specimen Evaluation

Following completion of the exposure duration, the surfaces of the reduced section of the intact 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, evidence of cracking is either:

- a) Fracture of the tensile test specimen in the reduced section; or
- b) Visual observation of cracks on the reduced section of the tensile test specimen at 10X magnification. Investigative techniques employing metallography, scanning electron microscopy, or mechanical testing may be used to determine whether cracks on the reduced section are evidence of EAC or not.

NOTE: For CRA materials, an assessment of the severity of any corrosion pitting can be made by determining the maximum pit depth and evaluating the shape of pits (see ASTM G46²⁷ / ISO 11463²⁸).

8.7.2 Evidence of cracking of the tensile test specimen outside of the reduced section shall invalidate the specimen.

8.7.3 Time-to-fracture may be recorded using electrical timers and microswitches.

8.8 Reporting of Test Results

8.8.1 Test report shall include the following information:

- a) Sample identification, specimen location, orientation, geometry, and finishing process;
- b) Load method (e.g., constant or sustained load);
- c) Test environment (e.g., solution chemistry, test gas, temperature, pressure, initial pH, and final pH);
- d) Applied stress; and
- e) Time-to-fracture and no-fracture data or the visual observation of surface cracks at the end of the test shall be reported for each specimen.

8.8.2 For qualification of materials in accordance with ANSI/NACE MR0175-2021/ISO 15156:2020, the chemical composition, heat treatment, mechanical properties, other information specified above, and data taken shall be reported, see report format in [Appendix D](#), [Tables D1](#) and [D2A](#).

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

9.1 Method B, the NACE Standard Three-Point Bent-Beam Test, provides for testing carbon and low-alloy steels subjected to tensile stress to evaluate resistance to cracking in low-pH aqueous environments containing H₂S. It evaluates EAC susceptibility of these materials in the presence of a stress concentration. The compact size of the three-point bent-beam test specimen facilitates testing small, localized areas and thin materials. Bent-beam test specimens loaded to a particular deflection give a cracking/no-cracking test result. When testing multiple test specimens at varying deflections, a statistically based critical stress factor (S_c) for a 50% probability of cracking 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.³²

NOTE: Method B is a three-point bent beam test listed as a product verification test in API 5CT⁴ primarily for thin wall tubing, refer to NACE TM0316³ regarding four-point bend testing.

9.1.1 This section sets forth the procedure for three-point bent-beam testing at ambient temperature and pressure. Special considerations for testing at non-ambient 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 evaluated for evidence of cracking. From the data obtained by testing multiple test specimens at varying deflections, a statistically based S_c for a 50% probability of cracking 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

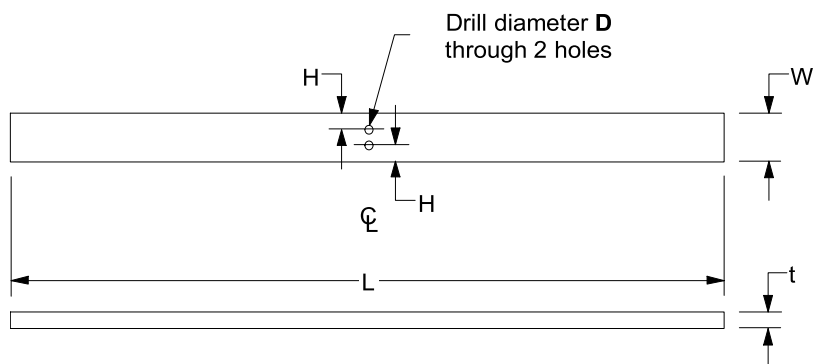
9.2.1 The dimensions of the standard three-point bent-beam test specimen shall be 4.57 ± 0.13 mm (0.180 ± 0.0050 in) wide, 1.52 ± 0.13 mm (0.060 ± 0.0050 in) thick, and 67.3 ± 1.3 mm (2.65 ± 0.050 in) long (see Figure 6). After machining, test specimens shall be stored in a low-humidity area, in a desiccator, 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 shall be reported with the test results.

9.2.3 The test specimen shall be fabricated in such a way as to avoid overheating, and to minimize any accidental cold working in the stressed section. Surfaces and edges of the test specimen shall be ground using 240 or higher grit emery paper parallel to the test specimen axis. The final average surface roughness ('Ra' value) shall be $0.81 \mu\text{m}$ (32 μin) or finer.

9.2.4 As shown in Figure 6, 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 ± 0.013	0.028 ± 0.0005 (No. 70 Drill)

Figure 6: Dimensional Drawing of the Standard Three-Point 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 each end on the compression side.

9.2.6 Test Specimen Cleaning – see [Paragraph 4.5](#).

9.3 Test Solution

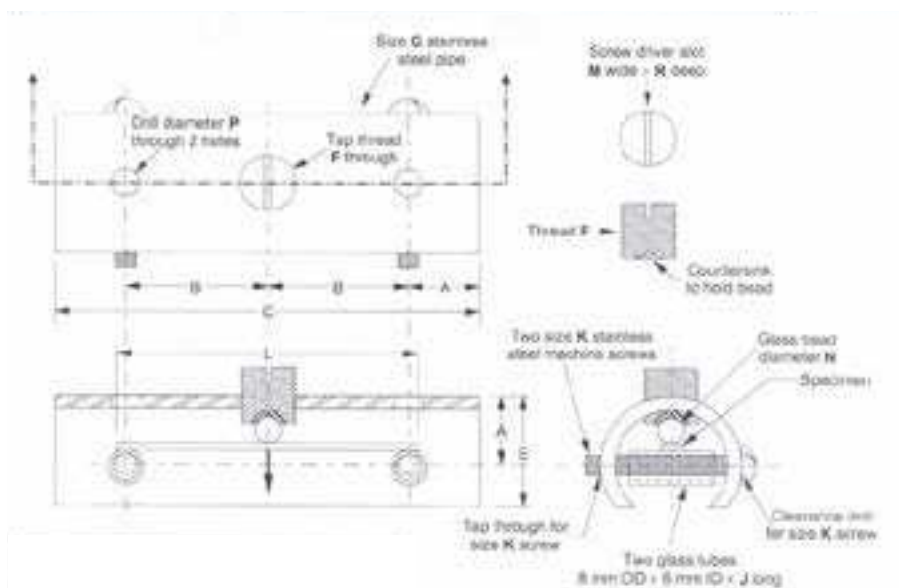
9.3.1 The test solution shall consist of 0.5 wt% glacial acetic acid dissolved in distilled or deionized water (e.g., 5.0 g [4.8 mL] of CH_3COOH dissolved in 995 g of distilled or deionized water). NaCl shall not be added to the test solution.

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.

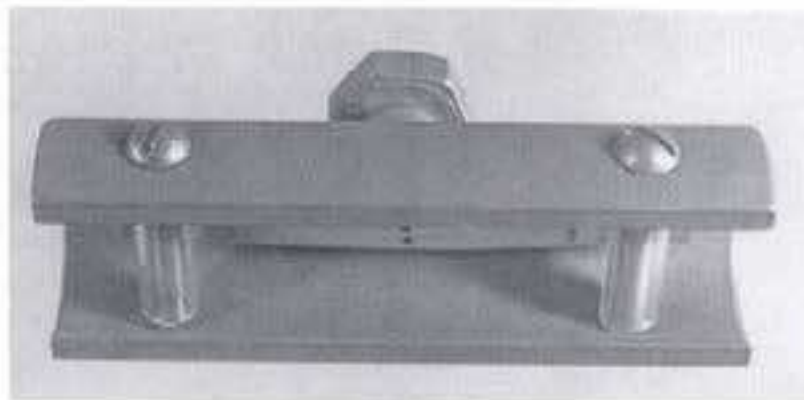
9.4.2 Tests shall be performed using constant-deflection fixtures that employ three-point bending of the test specimen. (See Figures 7[a] and 7[b]).



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 7: Typical Stressing Fixture for Three-Point 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 shall be electrically isolated from the test specimen.
- 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 minimum test solution volume of 20 mL/cm² of test specimen surface area.
 - 9.4.5.2** The test vessel shall be valved at both the inlet and outlet to prevent contamination of the test solution by oxygen.
 - 9.4.5.3** The test gas 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 (10⁴ 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.
 - 9.5.3** The test specimen deflection shall be calculated for each of the pseudo-stress values using Equation (5):

⁽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).

$$D = \frac{S\ell^2}{6Et} \quad (5)$$

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. The deflection should be carefully applied to avoid exceeding the desired value. If the desired deflection is exceeded by more than 2%, 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 and solution transfer requirements are given in [Paragraph 6.7](#).
- 9.6.5 The test solution shall then be saturated with H₂S at a rate of at least 100 mL/min for at least 20 min/L of test solution. The H₂S in the test vessel shall be replenished periodically by bubbling H₂S for a duration of 20 to 30 min at a rate of at least 100 mL/min per liter 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 the week.
- 9.6.6 The timing for the start of test is defined in [Paragraph 6.1.8](#).
- 9.6.7 The test shall be terminated after 720 hours or when all test specimens have failed, whichever occurs first.
- 9.6.8 Additional test specimens and iterative testing may be necessary to define the S_c closely.

9.7 Specimen Evaluation

- 9.7.1 The presence of cracking 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 Following exposure, the surface of the test specimens should be cleaned and visually inspected at 10X for evidence of cracking after bending through approximately 20 degrees by hand.

9.8 Reporting of Test Results

9.8.1 Test report shall include the following information:

- Sample identification, specimen location, orientation, geometry, and finishing process;
- Load method and applied load;
- Test environment (e.g., solution chemistry, test gas, temperature, pressure, start and end pH); and
- Cracking/no-cracking data and nominal outer fiber pseudo-stress (S) values shall be reported. Time-to-cracking data are optional.

9.8.2 The S_c shall be calculated using Equation (6) for S values expressed in MPa, or Equation (7) for S values expressed in psi:

$$S_c = \frac{\frac{\sum S}{68.95 \text{ MPa}} + 2\sum T}{n} \quad (6)$$

where:

S = nominal outer fiber pseudo-stress (in MPa) used to calculate the beam's deflection;

T = the test result (i.e., +1 for no-cracking and -1 for cracking); and

n = the total number of test specimens tested.

When Equation (6) is used, all pseudo-stress data that are more than ± 210 MPa from the initial calculated value $S_c \times 68.95$ MPa shall be discarded, and a new S_c value shall be recalculated. The recalculated S_c value eliminates low and high bias data.

$$S_c = \frac{\frac{\sum S}{10^4 \text{ psi}} + 2\sum T}{n} \quad (7)$$

where:

S = nominal outer fiber pseudo-stress (in psi) used to calculate the beam's deflection;

T = the test result (i.e., +1 for no-cracking and -1 for cracking); and

n = the total number of test specimens tested.

When using Equation (7), all pseudo-stress data that are more than $\pm 3.0 \times 10^4$ psi from the initial calculated value $S_c \times 10^4$ 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 Additionally, the chemical composition, heat treatment, mechanical properties, and other data taken may be reported, see report format in [Appendix D](#), [Tables D1](#) and [D2B](#).

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

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

- 10.1.1** This section sets forth the procedure for C-ring testing at ambient temperature and atmospheric pressure. Special considerations for testing at non-ambient 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 G38³³ 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 8.
- 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 may also be evaluated by C-ring test specimens. Using any finishing process other than machining shall be reported with the test data.
- 10.2.4.2** A small (typically 0.5 – 1.0 mm) fillet radius or 45-degree chamfer may be machined on the outside and/or inside edges of the C-ring specimen to reduce the potential for crack initiation in the more highly strained material at the specimen edges.

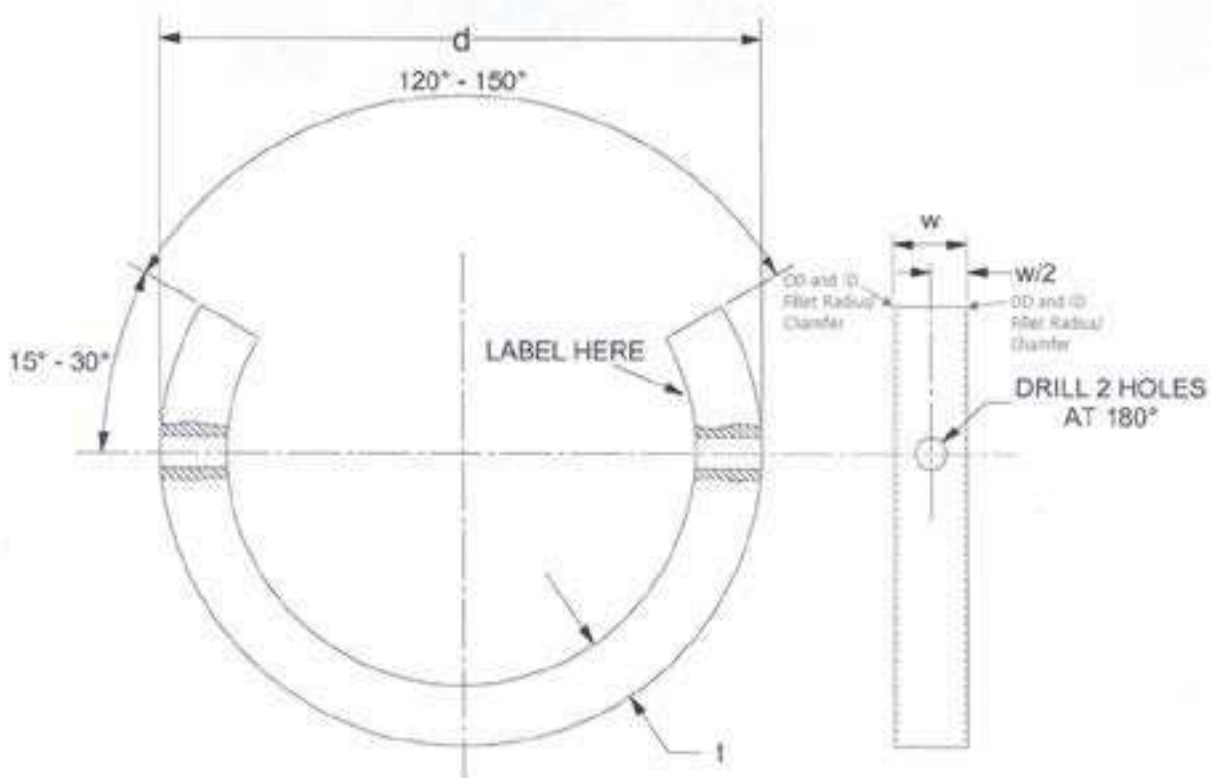


Figure 8: Dimensional Drawing of the C-Ring Test Specimen

10.2.5 Test Specimen Identification

Both ends of the C-ring specimen should be stamped or vibratory stenciled.

10.2.6 Test Specimen Cleaning – see [Paragraph 4.5](#).

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 supported such that only 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 minimum test solution volume of 20 mL/cm² of test specimen surface area.

- 10.4.3.2** The test gas bubbles should not impinge on the C-ring test specimens.

10.5 Deflection Calculations

The deflection shall be applied on the C-ring specimen using one of the two methods detailed below.

- 10.5.1** The deflection necessary to obtain the desired stress on the outer diameter of the C-ring test specimen can be calculated using Equation (8):

$$D = \frac{\pi d (d-t) S}{4tE} \quad (8)$$

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.

NOTE: This equation cannot be applied for stressing the inner diameter of the C-ring test specimen.

- 10.5.1.1** Deflections calculated by Equation (8) should be limited to stresses up to the material elastic limit. For many CRAs, the elastic limit is well below the 0.2% offset yield strength. 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 (8) may be used to calculate the deflection necessary to stress the test specimen to 100% of the 0.2% offset yield strength (S_y) by substituting $S_y + E (0.002)$ for S in the original equation. This relationship has been shown to be valid for a wide range of alloy systems, heat treatments, and test specimen dimensions. In all cases, the actual applied strain was found to significantly exceed the target strain associated with the 0.2% offset yield strength.³³

- 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 yield strength.

- 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 4.5](#).

NOTE: This approach may be applied for stressing the inner diameter of the C-ring test specimen.

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) or to the required strain value.
- 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 target deflection or strain is exceeded by more than 2%, the test shall be run at the higher load or discarded.
- 10.6.3** The C-ring test specimens shall be cleaned and placed into the test vessel.
- 10.6.4** Deaeration and solution transfer requirements are given in [Paragraph 6.7](#).
- 10.6.5** The test solution shall then be saturated with the test gas per the requirements of [Paragraph 6.8](#).
- 10.6.6** The timing for the start of the test is defined in [Paragraph 6.1.8](#).
- 10.6.7** The termination of the test shall be after 720 hours, or following cracking of all C-ring test specimens, whichever occurs first.

10.7 Specimen Evaluation

- 10.7.1** Highly stressed C-rings of alloys that are appreciably susceptible to EAC tend to fracture through the entire thickness or to crack in a way that is conspicuous. However, with more EAC-resistant alloys, cracking frequently begins slowly and it 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. 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 EAC can be differentiated from mechanically induced cracks by the corroded nature of the crack surface. C-rings may be evaluated for evidence of subsurface cracking by sectioning circumferentially, mid-width at the C-ring apex, followed by metallographic preparation of the cut faces.

NOTE: For CRA materials, an assessment of the severity of any corrosion pitting can be made by determining the maximum pit depth and evaluating the shape of pits (see ASTM G46²⁷ / ISO 11463²⁸).

10.8 Reporting of Results

- 10.8.1** Test report shall include the following information:
- a) Sample identification, specimen location, orientation, geometry, and finishing process;
 - b) Load method and applied load/strain;
 - c) Test environment (e.g., solution chemistry, test gas, temperature, pressure, initial solution pH, test start and final pH, and H₂S concentration [if specified]); and
 - d) Cracking/no-cracking data shall be reported for each specimen. If time-to-cracking data are recorded, they shall be reported.
- 10.8.2** For qualification of materials in accordance with ANSI/NACE MR0175-2021/ISO 15156:2020, the chemical composition, heat treatment, mechanical properties, and other data taken shall be reported, see report format in [Appendix D](#), [Tables D1](#) and [D2C](#).

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 EAC, expressed in terms of a critical stress intensity factor, K_{ISSC} for SSC and K_{EAC} for the more general case of EAC, 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. Method D gives a direct numerical rating of crack propagation resistance and does not depend on evaluation of cracking/no-cracking results.³⁴ The subject of fracture mechanics testing for evaluation of EAC resistance is currently under consideration by AMPP Standards Committee (SC) 08 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 ambient temperature and atmospheric pressure and enables computation of K_{ISSC} . When the special considerations set forth in Section 7 for testing at non-ambient temperature and pressure are specified, the computed stress intensity factor should be written as K_{EAC} . The equations needed to compute K_{EAC} 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 EAC.

11.2 Test Specimen

11.2.1 The standard DCB test specimen design shall be in accordance with Figure 10 (a). A double-tapered wedge shall be used to load the DCB test specimen (see Figure 10 [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. 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 10 (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 10 (a) may be considered. Subsize DCB test specimens of carbon and low-alloy steels give different K_{ISSC} values than the standard DCB test specimens. Acceptance criteria for subsize DCB specimens should be agreed upon prior to testing.

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 shall be machined or ground carefully to avoid overheating and cold working.

11.2.5 Fatigue precracking at the base of the chevron/EDM notch can facilitate crack initiation in testing low yield strength materials, and impede sidearm cracking in testing low K_{ISSC} materials where the H_2S partial pressure is reduced or the solution pH increased.³⁵ When fatigue precracking is used:

11.2.5.1 Fatigue precracking of the standard-size specimen should be conducted under load control within $\pm 5\%$ of the load at frequencies lower than 50 Hz. The ratio of minimum to maximum load shall be in the range of 0.1 to 0.2. Recommended precracking loads and load calculations based on the initial stress intensity factor imparted by the wedge are given in [Appendix E](#) (Nonmandatory). Displacement control instead of load control is permitted if the method is verified and documented, and the control within $\pm 5\%$ of the load is ensured.

- 11.2.5.2 The test machine load shall be calibrated in accordance with an appropriate standard, e.g., per ASTM E4.³⁶
- 11.2.5.3 The fatigue precracking load shall be either the lesser of 70% of the expected initial stress intensity factor (K_I) imparted by the wedge or 30 MPa√m (27 ksi√in).
- 11.2.5.4 The precrack should be sharpened at two-thirds of the maximum precracking load for at least 20,000 cycles to extend the crack through any zone of residual compressive stresses that might have developed.
- 11.2.5.5 Acceptable precrack lengths are in the range of 1 to 3 mm (0.04 to 0.12 in) past the base of the chevron/EDM notch.
- 11.2.5.6 The maximum precracking load and the actual precracking stress intensity factor shall be reported for each specimen.

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 Test Specimen Hardness Measurement

When specified by API 5CT, three or more hardness readings shall be taken on each DCB test specimen prior to testing. The average test specimen hardness value and test specimen face tested shall be identified as 'DCB test specimen hardness data for information only' and reported with the DCB 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 9) or on the broad faces near the side groove of thinner subsized specimens.

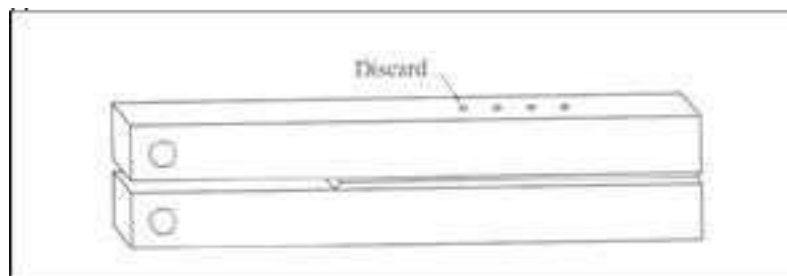


Figure 9: Location of Hardness Impressions on DCB Specimen

11.2.8 Dimensional Check

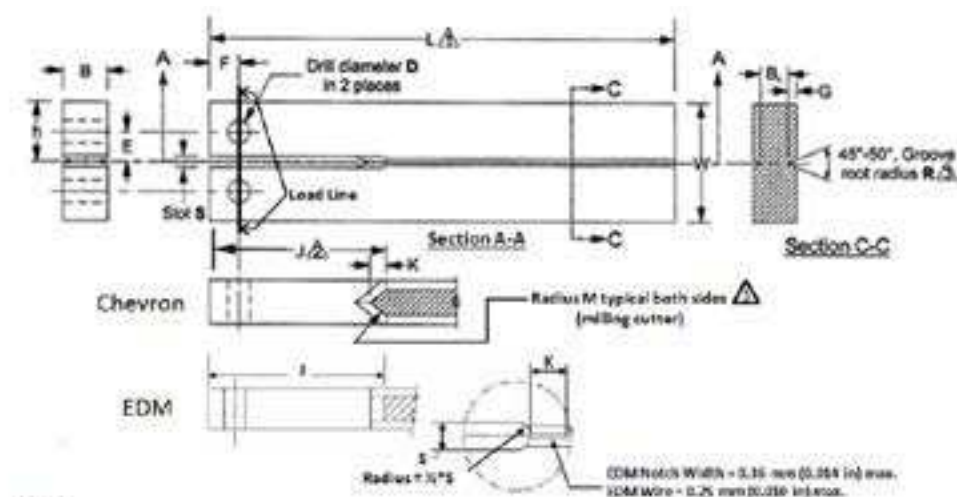
Dimensions B , B_n , $2h$, and the distance of the hole centers from the near end of the DCB test specimen shall be measured after cleaning (see [Paragraph 4.5](#)). A blade micrometer should be used for measuring B_n . Any values that lie outside the limits shown in [Figure 10 \(a\)](#) shall be recorded for later use in computing K_{ISSC} (see [Paragraph 11.6.3](#)).

11.2.9 Test Specimen Cleaning – see [Paragraph 4.5](#).

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 a minimum of 10 mL/cm² of test specimen area (generally, 1 L of test solution per DCB specimen is used).
- 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 the arms.
- 11.4.4** The test gas bubbles should not impinge on the DCB test specimens.



NOTES:

- 1 A DCB specimen of length L may be used if very low cracking resistance is expected.
- 2 A short electrodischarge machine (EDM) notch may substitute the Chevron crack starter.
- 3 Seams 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 ± 0.05 mm (0.188 ± 0.002 in)	2.86 ± 0.05 mm (0.113 ± 0.002 in)
6.35 ± 0.05 mm (0.250 ± 0.002 in)	3.81 ± 0.05 mm (0.150 ± 0.002 in)
12.7 ± 0.05 mm (0.500 ± 0.002 in)	7.62 ± 0.05 mm (0.300 ± 0.002 in)
Full Wall	$0.6 \cdot B$

Figure 10 (a): Dimensional Drawing of the DCB Specimen

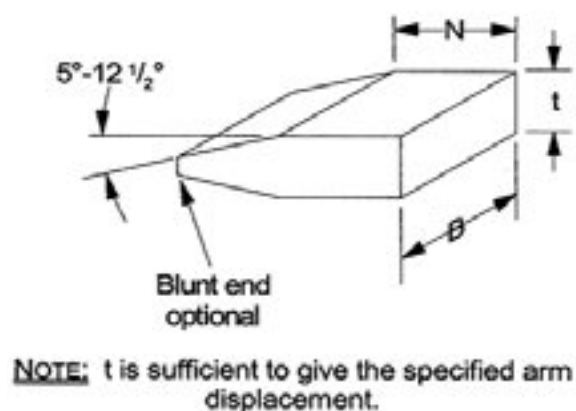


Figure 10 (b): DCB Specimen—Double-Tapered Wedge

11.5 Testing Sequence

11.5.1 Select wedges to achieve specified arm displacement.

11.5.1.1 Measure the slot thickness.

11.5.1.2 The arm displacement shall be carefully controlled because initial stress intensity affects the final K_{ISSC} .³⁷ The arm displacement shall not exceed the limits listed in Tables 1 and 2 when testing API and other grade tubular steels in Solution A and 100% H₂S and when testing C110 in Solution D with pH 4.0, and 7% H₂S/N₂. Table 3 provides guidance in choosing arm displacements for low-alloy steel products not listed in Table 1 and for other alloys when testing in Solution A and 100% H₂S. Other arm displacements may be used to determine K_{Limit} (see [Appendix F](#) [Nonmandatory]).

NOTE: For nonstandard products and fit-for-purpose testing, namely variation in H₂S concentration, 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).

11.5.1.2.1 When testing steels with yield strengths below 550 MPa (80 ksi), the arm displacement (δ) may be calculated using Equation (9a) or (9b), in which YS is yield strength:

$$\delta \text{ (mm)} = [2.02 - \frac{\text{YS (MPa)}}{530} + 0.13 \text{ mm}] \quad (9a)$$

$$\delta \text{ (in)} = [0.080 - \frac{\text{YS (ksi)}}{1,953} + 0.0050 \text{ in}] \quad (9b)$$

Table 1
Arm Displacements for API and Other Grade Oilfield Tubular Steels in Solution A and 100% H₂S

Grade ^(A)	Yield Strength Range		Nominal Arm Displacement (δ)	
	MPa	(ksi)	mm	(in)
			Tolerances: +0.03/-0.05	Tolerances: +0.001/-0.002
L80	552-655	(80-95)	0.84	(0.033)
C90	621-724	(90-105)	0.75	(0.030)
T95	655-758	(95-110)	0.71	(0.028)
R95	655-758	(95-110)	0.71	(0.028)
Grade 100 ^(B)	689-793	(100-115)	0.64	(0.025)
Grade 105 ^(B)	724-827	(105-120)	0.58	(0.023)
C110	758-827	(110-120)	0.51	(0.020)
P110	758-965	(110-140)	0.51	(0.020)
Q125	862-1,030	(125-150)	0.42	(0.017)
Other HSS grades	>862	(>125)	0.42	(0.017)

^(A) API grades unless noted otherwise.

^(B) Non-API grades.

Table 2
Arm Displacement for API 5CT Grade C110 in Solution D with pH 4.0 and 7% H₂S/N₂

Grade ^(A)	Yield Strength Range		Nominal Arm Displacement (δ)	
	MPa	(ksi)	mm	(in)
			Tolerances: +0.03/-0.05 Tolerances: +0.001/-0.002	
C110	758-827	(110-120)	0.89	(0.035)

^(A) API grades unless noted otherwise.

Table 3
Suggested Arm Displacement Ranges for Selected Alloys and Strength Levels in Solution A and 100% H₂S

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 1.

11.5.1.2.2

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 10 \[a\]](#)). 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.

11.5.1.2.3

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.

11.5.1.2.4

Press the wedge into the slot and flush with the end of the DCB test specimen. If a load fixture is used to spread the DCB arms, the maximum arm displacement shall be less than the actual wedge displacement.

NOTE: Overspreading of the DCB arms may result in plastic deformation or a zone of residual compressive stresses that may inhibit crack propagation.

11.5.1.2.5 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 line described in Paragraph 11.5.1.2.2. For the SSC-resistant carbon and low-alloy steels in Tables 1 – 3, 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 notch or fatigue precrack shall be extended through the zone of plastically deformed material before reloading the DCB test specimen.

- 11.5.2** Clean the specimen (see [Paragraph 4.5](#)).
- 11.5.3** Place the DCB test specimens in the test vessel. The test specimen shall be exposed to H₂S within 24 hours of wedge insertion.
- 11.5.4** Deaeration requirements for the test solution, test vessel, and after solution transfer are given in [Paragraph 6.7](#).
- 11.5.5** The test solution shall then be saturated with H₂S per the requirements of [Paragraph 6.8](#).
- 11.5.6** The timing for the start of the test is defined in [Paragraph 6.1.8](#).
- 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 ambient temperature.
- 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 Solutions C and 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, and/or low H₂S environments, it may be necessary to test for at least 21 days or longer. The duration of the test shall be specified by the user or purchaser and shall be reported with the test 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 errors that are introduced during the measuring process.³⁸ The lift-off load shall be determined within three days after exposure.
- 11.5.10.1** The test machine load shall be calibrated per ASTM E4.³⁶ 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).

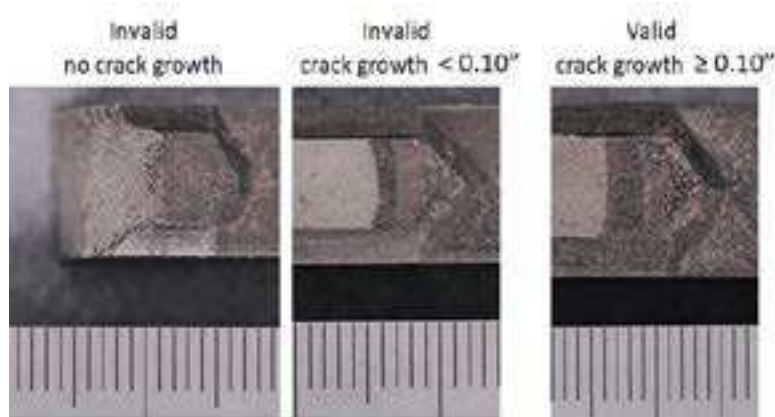
11.5.10.3 When determining the equilibrium wedge-load (P), the resolution of the load on the graph or digital display shall allow load determination within $\pm 1.0\%$ of the final wedge-load (P).

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.

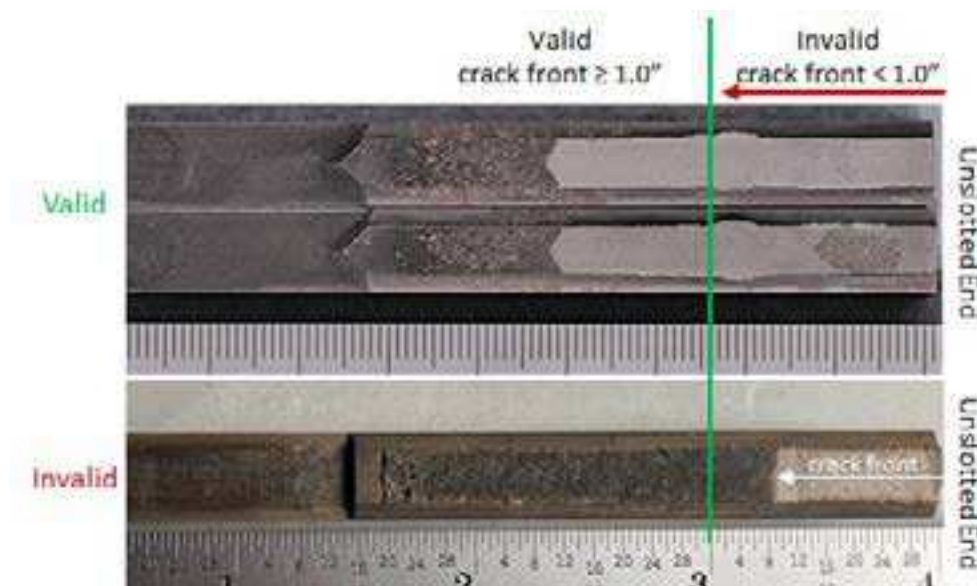
11.6 Determination of K_{ISSC}

11.6.1 The fracture surface shall be examined for the following characteristics as shown in Figure 11 (a-h):

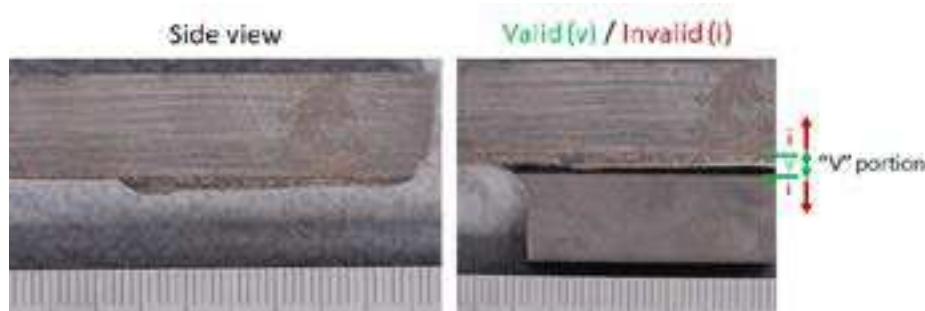
- (a) The fracture surface shall show at least 2.5 mm (0.10 in) crack growth beyond the base of the chevron, EDM notch, or fatigue precrack;



- (b) The crack front shall 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 negligible deviation shall not invalidate the test.

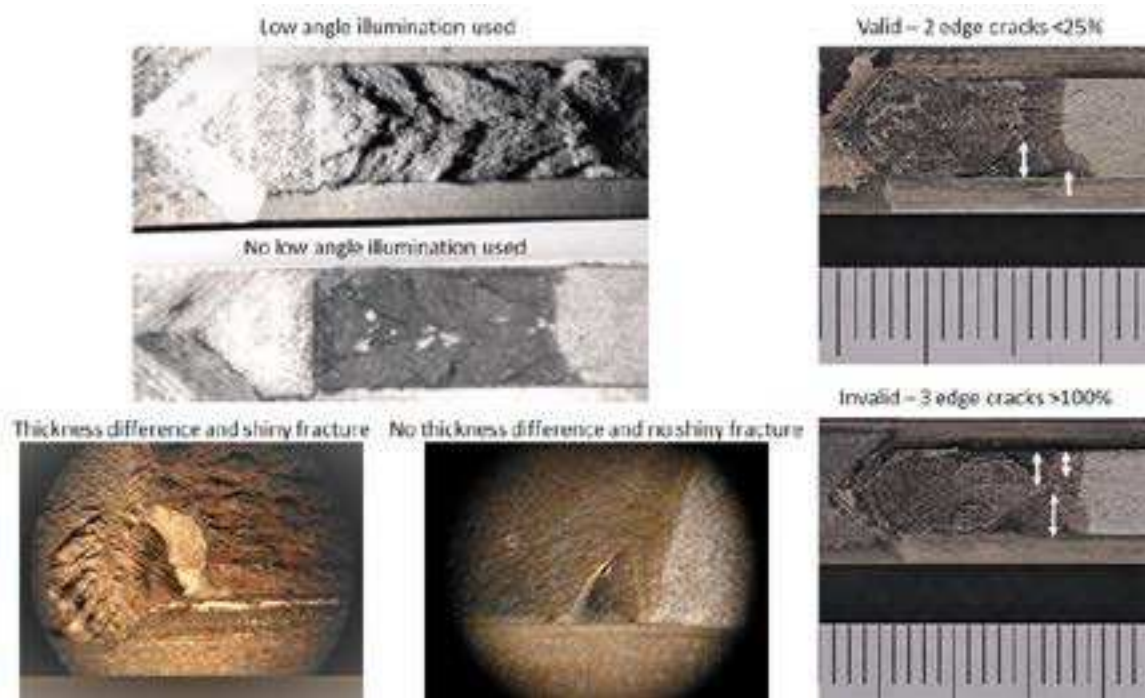


- (d) The crack front shall not be pinned; causes of pinning include internal cracks and fissures;

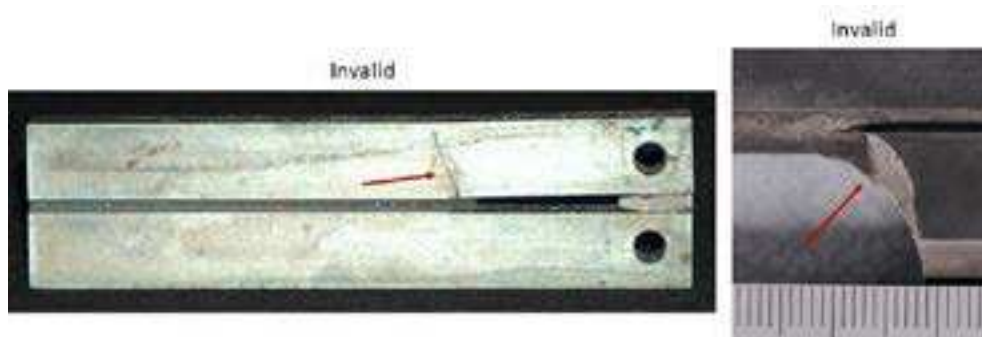


- (e) The fracture surface shall be free of significant thickness disturbance starting from the edge of the side grooves, so-called edge crack(s) (“shark’s tooth” or “leaf spring”).³⁹ The presence of edge cracks can be evaluated using low-angle illumination to reveal the thickness difference and presence of a shiny fracture. The width of edge crack(s) at the crack front on the fracture surface of a DCB specimen shall not exceed 25% of the web thickness (B_n) for a valid test. The summation of the width of edge crack(s) away from the crack front (formed earlier in the test) on a DCB specimen shall not exceed 100% of the B_n for a valid test. For a DCB test specimen 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 B_n for a valid test.

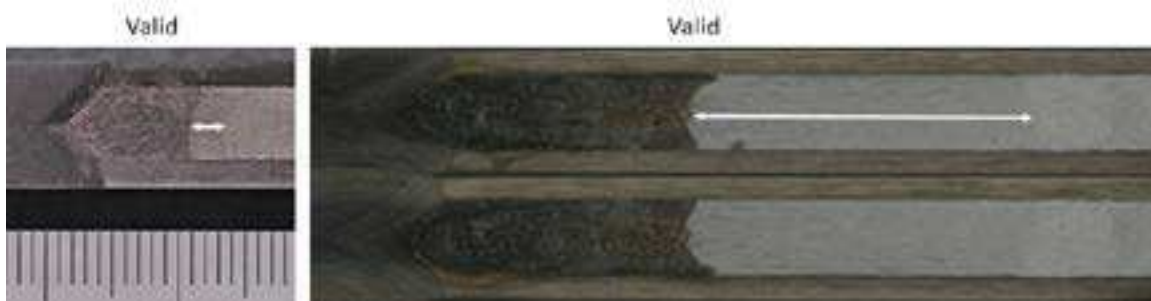
NOTE: Edge cracks may not affect results in the mechanical assurance curves (see Appendix G [Mandatory]).



- (f) The crack shall 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. 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 shall not lead on the edge of the fracture surface closer to the tubular internal surface.

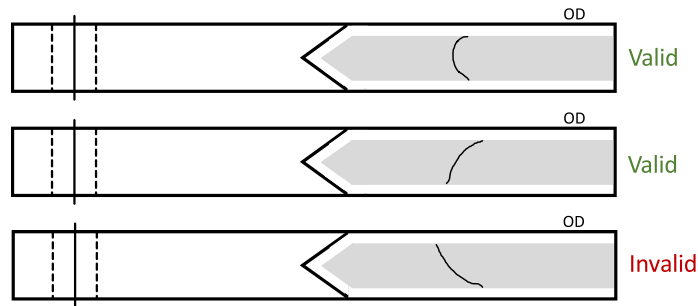


Figure 11: Fracture Surface Characteristics

- 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 (10):

$$K_{ISSC} \text{ (flat DCB test specimen)} = \frac{Pa(2\sqrt{3} + 2.38h/a)(B/B_n)^{1/3}}{Bh^{3/2}} \quad (10)$$

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 = 1,873 N (421 lbf);
a = 46.48 mm (1.830 in); and

Result:
 $K_{ISSC} = 35.3 \text{ MPa}\sqrt{\text{m}}$ (32.1 ksi $\sqrt{\text{in}}$).

11.6.4 The stress intensity factor for SSC of full-wall thickness (curved) DCB test specimens shall be computed using Equation (11):⁴⁰

$$K_{ISSC} \text{ (curved DCB test specimen)} = \left(\frac{3I}{Bh^3} \right)^{1/2} K_{ISSC} \text{ (flat DCB test specimen)} \quad (11)$$

where:

K_{ISSC} (flat DCB test specimen) is calculated using Equation (10); and

Quantity I is computed from Equation (12) as follows:

$$I = -\frac{h}{4} \left(\frac{D^2}{4} - h^2 \right)^{3/2} + \frac{D^2 h}{32} \left(\frac{D^2}{4} - h^2 \right)^{1/2} + \frac{D^4}{128} \sin^{-1} \left(\frac{2h}{D} \right) + \frac{h}{4} \left[\left(\frac{D}{2} - B \right)^2 - h^2 \right]^{3/2} \\ - \frac{h}{8} \left(\frac{D}{2} - B \right)^2 \left[\left(\frac{D}{2} - B \right)^2 - h^2 \right]^{1/2} - \frac{1}{8} \left(\frac{D}{2} - B \right)^4 \sin^{-1} \left(\frac{2h}{D - 2B} \right) \quad (12)$$

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 percent 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:
 $K_{ISSC} = 35.4 \text{ MPa}\sqrt{\text{m}}$ (32.2 ksi $\sqrt{\text{in}}$).

11.7 Reporting of Test Results:

11.7.1 For each sample, the following data shall be reported:

- Sample identification, specimen identification and location, orientation, geometry, and finishing process;
- Actual specimen dimension: height (h), thickness (B), web thickness (B_n), crack length (a), arm displacement (δ), lift-off load (P);
- Test environment (e.g., solution chemistry, test gas, temperature, pressure, initial, start, and final pH, start and final H_2S concentration [if specified]);
- Test duration;

- e) HRC hardness results (if specified);
- f) Individual K_{ISSC} values of valid tests and invalidation reason (see [Paragraph 11.6.1](#)) of invalid tests;
- g) Average K_{ISSC} value and standard deviation of a minimum of three valid DCB specimens;
- h) Fatigue precrack intensity factor and maximum load, if requested.

- 11.7.2** For qualification of materials in accordance with ANSI/NACE MR0175-2021/ISO 15156:2020, the chemical composition, heat treatment, mechanical properties, other information specified above, and data taken shall be reported, see report format in [Appendix D](#), [Tables D1](#) and [D2D](#).
- 11.8** Quality assurance methods of evaluating the final mechanical state and validity within the test parameters of each DCB test specimen are in [Appendix G](#) (Mandatory).
- 11.9** A recommended method for determining K_{LIMIT} and $K_{applied}$ is provided in Appendix F. K_{LIMIT} represents the crack initiation toughness for a specific material tested in a specific environment and estimates the minimum K_{ISSC} . $K_{applied}$ is the applied stress intensity factor obtained after wedge insertion and before the environmental exposure.

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⁽⁷⁾ Occupational Safety and Health Administration (OSHA), U.S. Department of Labor, 200 Constitution Ave. NW, Washington, DC 20210, www.osha.gov.

⁽⁸⁾ American Chemistry Council (ACC) (formerly known as the Manufacturing Chemists Association [MCA] and then as the Chemical Manufacturers Association), 700 Second St. NE, Washington, DC 20002, www.americanchemistry.com.

Appendix A

Safety Considerations in Handling H₂S 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.

H₂S is perhaps responsible for more industrial poisoning accidents than any other single chemical. A number of these accidents have been fatal. H₂S must be handled with caution and any experiments using it must be planned carefully. The OSHA maximum allowable concentration of H₂S in the air for an eight-hour workday is 10 parts per million (ppm), well above the level detectable by smell.⁴¹ The olfactory nerves can become deadened to the odor after exposure of 2 to 15 min, depending on concentration, so odor is not a completely reliable alarm system.

Briefly, the following are some of the human physiological reactions to various concentrations of H₂S. 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 H₂S can be obtained from the Chemical Safety Data Sheet SD-36⁴² and from *Dangerous Properties of Industrial Materials*.⁴³

Fire and Explosion Hazards

H₂S is a flammable gas and yields poisonous sulfur dioxide (SO₂) 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 H₂S. The H₂S 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 H₂S 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 H₂S flow is interrupted. Suitable safety equipment shall be used when working with H₂S.

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 H₂S 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 should 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 H₂S 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 H₂S 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 EAC) 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 EAC, 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 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 H₂S 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 H₂S concentration in the test solution by iodometric titration.

Test Equipment

10 mL and 25 mL 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 (Na₂S₂O₃) 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 the determination of the test solution H₂S concentration, for a series of H₂S partial pressure ranges from 100 kPa to less than 1 kPa at ambient temperature. 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 H₂S partial pressure.

Table C1
Titration Parameters for Determination of the Test Solution H₂S Concentration

pH₂S (kPa)	Minimum cH₂S (mg/L)	Test Solution Sample Volume (mL)	Iodine / Na₂S₂O₃ Concentration (N)
40 – 100	920 – 2,300	10	0.1
16 – 40	370 – 920	25	0.1
8.0 – 20	185 – 460	50	0.1
4.0 – 10	92 – 230	100	0.1
1.6 – 4.0	37 – 92	25	0.01
0.8 – 2.0	18 – 46	50	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 it 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 the solution samples.

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 H₂S concentration (mg/L) using Equation (C1):

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

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)

$$17,040 = \frac{34.08 \left(\frac{\text{g}}{\text{mole}} \text{H}_2\text{S} \right) \times 1,000 \left(\frac{\text{mg}}{\text{g}} \right)}{2 \left(\frac{\text{equivalents}}{\text{mole H}_2\text{S}} \right)}$$

Appendix D
Recommended Reporting Template for ANSI/NACE MR0175-2021/ISO
15156:2020 Material Qualification
(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 reporting format for material testing for all four methods (Method A, Method B, Method C, and Method D). The provided tables may not be sufficient to capture additional ANSI/NACE MR0175-2021/ISO 15156:2020 requirements for the reporting of objectively verifiable test data, particularly for testing conducted under non-ambient conditions. See [Section 7](#).

Table D1 is used to provide information about the tested material, including chemistry, heat treatment, product form, etc.

Tables D2 (A, B, C, and D) are used to provide the test SSC or SCC test result for each test Method (A, B, C, and D, respectively).

Table D1
NACE Uniform Material Testing Report Form (Part 1):
Testing in Accordance with NACE Standard TM0177^(A)

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 shall 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, pre-strain.

Table D2A
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 _____ ☐ Reduced Section Length _____ ☐ Finishing Process _____

☐ Constant Load ☐ Dead Weight ☐ Hydraulic ☐ Other _____

☐ Sustained Load ☐ Proof Ring ☐ Post-Test Proof Ring Deflection Measurement ☐ Spring ☐ Other _____

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

Test Gas Composition as Specified in 6.1.3 _____ ☐ Outlet Trap to Exclude Oxygen

Temperature: ☐ Maintained 24° ± 3 °C (75° ± 5 °F) ☐ Maintained _____ ±3 °C (±5 °F) ☐ Maintained _____ ±5 °C (±9 °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)			Target dissolved H ₂ S concentration	Visual Observations (surface fissuring, pitting, or other notable features)
	Location ^(B)	Orientation ^(C)	Yield Strength ^(D) ()	Ultimate Tensile Strength ()	Elongation (%)	Reduction in Area (%)	Hardness ()		Initial	Start	Final	Resource for dissolved H ₂ S target concentration	
								Time-to-Fracture (Hours) NF = No Fracture at 720 hours				Measured dissolved H ₂ S concentration	

^(A) Test method shall 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 shall 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 non-failed tensile test specimen at highest stress if summarizing data.

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Table D2B
NACE Uniform Material Testing Report Form (Part 2):
Testing in Accordance with NACE Standard TM0177 Method B—NACE Standard Three-Point 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 _____
 Test Gas Composition as Specified in 6.1.3 _____ ☐ Outlet Trap to Exclude Oxygen
 Temperature: ☐ Maintained $24^\circ \pm 3^\circ \text{C}$ ($75^\circ \pm 5^\circ \text{F}$) ☐ Maintained _____ $\pm 3^\circ \text{C}$ ($\pm 5^\circ \text{F}$) ☐ Maintained _____ $\pm 5^\circ \text{C}$ ($\pm 9^\circ \text{F}$)

	Test Specimen Properties							Pseudo-Stress (S) Value ()	S_c Value	Test Solution pH ^(E)		Target dissolved H_2S concentration	Visual Observations (surface fissuring, pitting, or other notable features)
Material Identification	Location ^(B)	Orientation ^(C)	Yield Strength ^(D) ()	Ultimate Tensile Strength ()	Elongation (%)	Reduction in Area (%)	Hardness ()			Start	Final	Resource for dissolved H_2S target concentration	
								Time-to-Fracture (Hours) NF = No Fracture at 720 hours				Measured dissolved H_2S concentration	

^(A) Test method shall 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 shall 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 non-failed bent-beam test specimen at highest stress if summarizing data.

Table D2C
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 Equipment: ☐ Bolting Material Same as Specimen ☐ Correction for Yield Applied

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

Test Gas Composition as Specified in 6.1.3 _____ ☐ Outlet Trap to Exclude Oxygen

Temperature: ☐ Maintained 24° ± 3 °C (75° ± 5 °F) ☐ Maintained _____ ±3 °C (±5 °F) ☐ Maintained _____ ±5 °C (±9 °F)

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

Test Specimen Properties								Applied Stress (% of Yield Strength)	Test Solution pH ^(E)			Target dissolved H ₂ S concentration	Visual Observations (surface fissuring, pitting, or other notable features)
Material Identification	Location ^(B)	Orientation ^(C)	Yield Strength ^(D) ()	Ultimate Tensile Strength ()	Elongation (%)	Reduction In Area (%)	Hardness ()		Initial	Start	Final	Resource for dissolved H ₂ S target concentration	
								Time-to-Fracture (Hours) NF = No Fracture at 720 hours				Measured dissolved H ₂ S concentration	

^(A) Test method shall 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 shall 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 non-failed C-ring test specimen at highest stress if summarizing data.

Table D2D
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_____
Test Gas Composition as Specified in 6.1.3_____ ☐ Outlet Trap to Exclude Oxygen
Target dissolved H₂S concentration_____ Resource for dissolved H₂S target concentration_____
Measured dissolved H₂S concentration_____
Temperature: ☐ Maintained 24° ± 1.7 °C (75° ± 3 °F) ☐ Maintained _____ ± 1.7 °C (± 3 °F)
For elevated temperature tests: ☐ Test vessel heated before test gas ☐ Test gas admitted before test vessel heated

	DCB Test Specimen Properties ^(D)							Data for Valid Tests												Test Solution pH ^(E)			Visual Observations (surface fissuring, pitting, or other notable features)		
Material Identification	Location ^(B)	Orientation ^(C)	Yield Strength ^(D) ()	Ultimate Tensile Strength ()	Elongation (%)	Reduction in Area (%)	Hardness ()	Arm Displacement (δ) ()				K _{precracking} () /Max. Load ()				K _{ISSC} or K _{IEAC} ()						Initial		Start	Final
								1	2	3	4	1	2	3	4	1	2	3	4	Mean	Std. dev.				

- ^(A) Test method shall 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 shall 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 non-failed DCB test specimen at highest stress if summarizing data.

Appendix E

Recommendations for Fatigue Precracking of DCB Specimens for Method D (DCB Test) (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 fatigue precracking load is either the lesser of 70% of the expected initial stress intensity factor (K_I) imparted by the wedge or $30 \text{ MPa}\sqrt{\text{m}}$ ($27 \text{ ksi}\sqrt{\text{in}}$).

Table E1 gives the maximum precracking loads satisfying the above condition for selected target arm displacements (minus tolerance) listed in [Table 1](#) of Section 11 for standard-size DCB specimens ($B = 9.53 \text{ mm}/0.375 \text{ in}$).

The maximum precracking load can be calculated from the initial crack length ($a_i = 35.51 \text{ mm}$ [1.398 in]) instead of the final crack length (a_f), using Equation (F1) ([Appendix F](#)).

When using a DCB specimen size other than standard size, multiply the loads given in Table E1 using the factor included in Table E2. The factors are based on the assumption that the compliance equation is not significantly different for non-standard size specimens).

Table E1
Maximum Precracking Loads and Stress Intensity Factors for Standard-Size DCB Specimen

Arm Displacement, mm (0.001 in)*	Maximum Load N (lbf)	$K_{I \text{ precracking}}$ $a_i: 35.51 \text{ mm}/(1.398 \text{ in})$ $\text{MPa}\sqrt{\text{m}}$ ($\text{ksi}\sqrt{\text{in}}$)
>0.66 (>26)	1,926 (433)	29.0 (26.4)
0.59 (23)	1,926 (433)	29.0 (26.4)
0.53 (21)	1,926 (433)	29.0 (26.4)
0.46 (18)	1,334 (300)	19.5 (17.7)
<0.46 (<18)	899 (202)	13.5 (12.3)

*Nominal selected arm displacement of Table 1 minus tolerance.

Table E2
Factor to Calculate the Maximum Precracking Load for All DCB Specimen Sizes

B mm (in)	Factor to Calculate Maximum Precracking Load with Respect to the Standard-size Specimen
12.7 (0.500)	1.332
9.53 (0.375)	1.000
6.35 (0.250)	0.666
4.76 (0.188)	0.499

The maximum precracking load and the actual precracking stress intensity factor shall be reported for each specimen. The actual precracking stress intensity factor shall be calculated by using Equation (E1).

$$K_{I \text{ precracking}} = \frac{P_{I \text{ precracking}} a_i (2a\sqrt{3} + 2.38 \frac{h}{a_i}) (B/B_n)^{1/3}}{B h^{3/2}} \quad (\text{E1})$$

where:

$K_{\text{precracking}}$ = precracking stress intensity factor;

a_f = final fatigue precrack length;

$P_{\text{precracking}}$ = maximum load used for precracking procedure; and

h, B, B_n = DCB specimen parameters as defined in [Figure 10](#).

The final fatigue precrack length determined after opening the specimens is equal to the initial crack length used for determination of K_{applied} . The precrack length shall be measured; see [Paragraph 11.6.2](#).

Precracked DCB specimens should be stored in a non-humid environment, preventing the deterioration of the crack tip. Sharpening of the crack prior to inserting the specimens into the vessel is permitted if both the final crack length is not exceeded and the precracking load is maintained within $\pm 5\%$.

Other documented and validated fatigue precracking procedures may be used when agreed upon between the user and purchaser.

Appendix F

Recommended Method for Determining K_{applied} and K_{LIMIT} for the Method D (DCB) Test⁴⁴ (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_{applied} 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_{applied} . The use of K_{LIMIT} as a parameter for design is under study.

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

Determination of K_{LIMIT} :

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 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 [Paragraph 11.6](#); and
 K_{applied} as defined in the following paragraph.

Determination of K_{applied} :

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). For the chevron DCB test specimen, use the effective starting crack of 30.02 mm (1.182 in) for any K_{applied} or K_{LIMIT} calculations.

The following procedure shall be used to determine K_{applied} 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 using Equation (G4) for flat DCB test specimens.

The calculated initial load, P_i , shall be calculated by using Equation (F1) for flat DCB test specimens:

$$P_i = \delta_f EB / (-26.232 + 51.866 a_i/h + 8.523 (a_i/h)^2 + 8.5179 (a_i/h)^3) \quad (\text{F1})$$

The K_{applied} of flat DCB test specimens shall be calculated using Equation (F2):

$$K_{\text{applied}} (\text{flat DCB test specimen}) = \frac{P_i a_i (2 \sqrt{3} + 2.38 h/a_i)(B/B_n)^{1/3}}{B h^{3/2}} \quad (\text{F2})$$

where:

B , B_n , and h have values in accordance with [Paragraph 11.6.3](#);

δ_i = 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_{applied} , plot the line of $K_{\text{ISSC}} = K_{\text{applied}}$. Then plot the K_{ISSC} versus K_{applied} 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 $K_{\text{ISSC}} = K_{\text{applied}}$ line. The intersection is defined as K_{LIMIT} . See Figure F1 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 (F3):

$$K_{\text{ISSC}} = m K_{\text{applied}} + c \quad (\text{F3})$$

where:

m = slope of line; and

c = intercept of the line.

The K_{LIMIT} is calculated by using Equation (F4):

$$K_{\text{LIMIT}} = c / (1 - m) \quad (\text{F4})$$

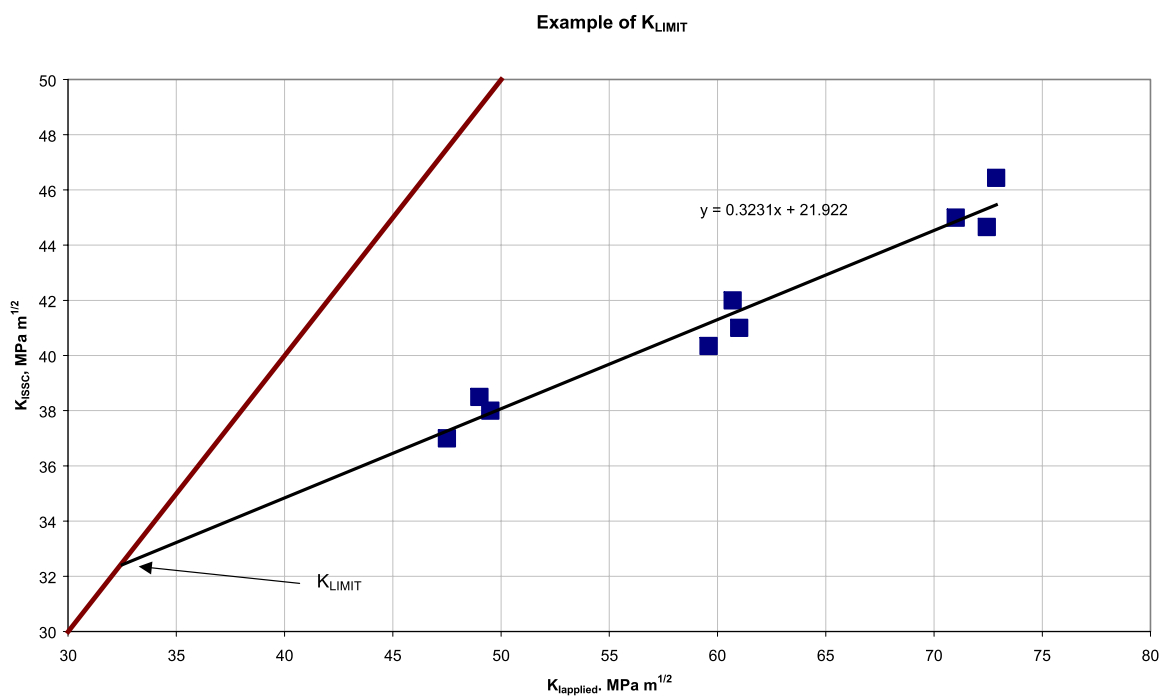


Figure F1: Example of K_{LIMIT} Determination

Appendix G

Determination of Mechanical Quality Assurance of Test Results for Method D (DCB Test)⁴⁵ (Mandatory)

The purpose of this appendix is to provide the quality assurance methods for evaluating the final mechanical state of the NACE TM0177 Method D (DCB) test specimen. They show that the DCB test results were within the parameters of the test method after environmental exposure. Both methods (G1 and G2) shall be performed, as each method examines different variables in the testing for validity.

The following information is required for each DCB test:

For each material/material grade (for suite of tests):

$\delta_{\min}$ = minimum allowable arm displacement minus 0.0254 mm (0.001 in);
 $\delta_{\max}$ = maximum allowable arm displacement plus 0.0254 mm (0.001 in); and
 E = measured elastic modulus or default of 2.07×10^5 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 the user and purchaser how to proceed with the limits for mechanical compliance analysis.

For each DCB test specimen:

P = measured lift-off load;
 a_f = measured final crack length;
 B = specimen thickness;
 h = arm height ($\frac{1}{2}$ specimen width); and
 δ_m = measured arm displacement before the test start.

G1 Graphing Method Procedure

On a graph of a_f/h versus P/B , plot curves of constant $\delta_{\max}$ and constant $\delta_{\min}$ using Equation (G1).

$$P/B = \delta_m E / (-26.232 + 51.866 a_f/h + 8.523 (a_f/h)^2 + 8.5179 (a_f/h)^3) \quad (G1)$$

where:

δ_m is $\delta_{\max}$ or $\delta_{\min}$ and
 $(P/B, a_f/h)$ are points on the curve of $\delta_{\max}$ or $\delta_{\min}$

Each DCB test result should be recorded as a point of $(P/B, a_f/h)$ on the graph. Equation (G1) is valid for both chevron and EDM notch.

Plotting a Minimum K_{ISSC} Criterion (Optional)

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 (G2):

$$P/B = K_{\text{ISSC}} h^{1/2} / ([2\sqrt{3} a_f/h + 2.38] [B/B_n]^{1/3}) \quad (G2)$$

Evaluation

If the DCB test specimen crack surface is valid, then test results plotted between the $\delta_{\max}$ and $\delta_{\min}$ 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 G1 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:

- Incorrect DCB dimensions measurements
- Machining
- Improper wedge placement
- Wrong arm displacement used or measured
- Invalid crack plane (nonplanar fracture)
- Lift-off load or final crack length improperly measured (e.g., missing dry crack length)
- Wrong modulus used
- Mixing of specimens

In the 'Optional' region of Figure G1, the DCB test result may be deemed to be valid. This test condition is considered more severe than the standard test and therefore indicates that the material is resistant.⁴⁵

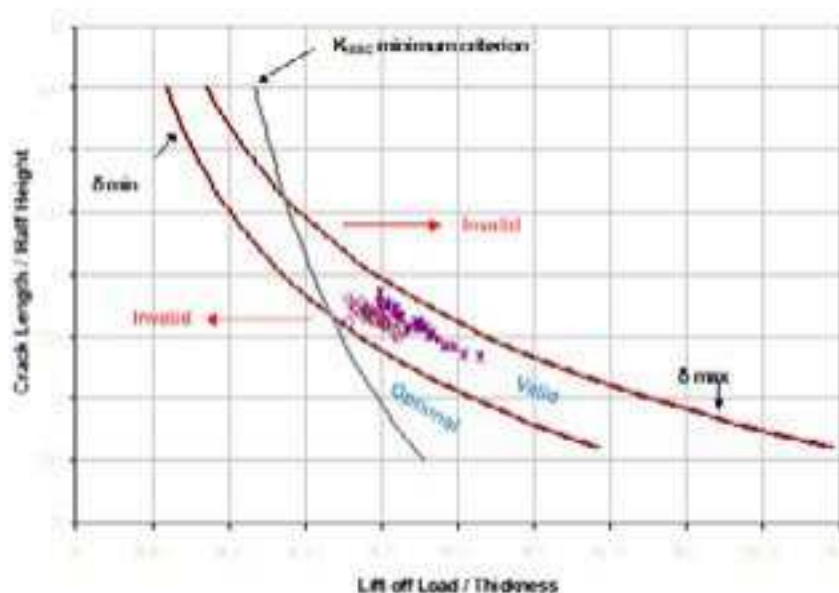


Figure G1: Example of Quality Assurance Plot

G2 Elastic Compliance Method Procedure

For a given crack length, the load applied for separating arms is proportional to the arm displacement, this is the stiffness of the specimen, which is the reciprocal of the specimen compliance C . The compliance of the DCB specimen has been determined as a function of crack length, arm height, and specimen thickness (Equation [G3]).⁴⁶

$$C = \delta_m/P = (-26.232 + 51.866 a/h + 8.523 (a/h)^2 + 8.5179 (a/h)^3)/(EB) \quad (G3)$$

Equation G3 is valid for both chevron and EDM notch. However, in cases where a generalized plasticity is developing at the crack tip, this equation is no longer valid.⁴⁷ It may occur for low yield strength materials and/or when large arm displacement is applied.

To verify the mechanical quality assurance of the DCB test results, compare the measured arm displacement at the start (δ_m) with the arm displacement calculated at the end of the test (δ_i) using compliance Equation (G4).

$$\delta_i = P (-26.232 + 51.866 a/h + 8.523 (a/h)^2 + 8.5179 (a/h)^3)/EB \quad (G4)$$

A difference between calculated and measured arm displacements greater than ± 0.0508 mm (0.002 in) shall be documented.

Displacement tolerance should be agreed upon between the user and purchaser.

Figure G2 shows an example graph of a mechanical quality assurance of measured arm displacement versus calculated arm displacement using Equation (G4). The example DCB data are within the lower and upper tolerance limits.

NOTE: Typical causes of data being outside of the tolerance are incorrect final lift-off load value and/or incorrect value of final crack length (e.g., dry crack not included in the final crack length measurement).

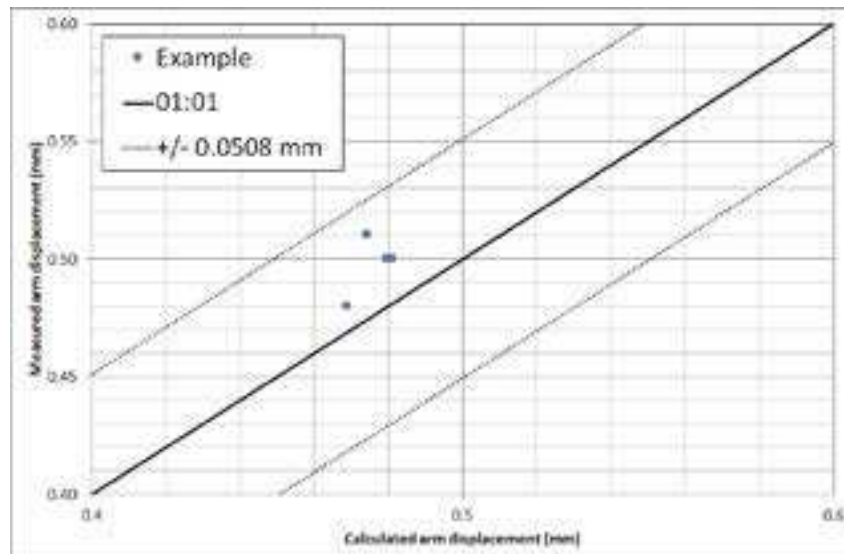


Figure G2: Example of Elastic Compliance of DCB Specimen