

## **Standard Test Method**

# **Laboratory Testing of Metals for Resistance to Sulfide Stress Cracking and Stress Corrosion Cracking in H<sub>2</sub>S Environments**

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## Foreword

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

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

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

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

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

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

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

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

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

## NACE International Standard Test Method

# Laboratory Testing of Metals for Resistance to Sulfide Stress Cracking and Stress Corrosion Cracking in H<sub>2</sub>S Environments

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## Section 1: General

1.1 This standard covers the testing of metals subjected to tensile stresses for resistance to cracking failure in low-pH aqueous environments containing H<sub>2</sub>S. Carbon and low-alloy steels are commonly tested for EC resistance at room temperature where SSC susceptibility is typically high. For other types of alloys the correlation of EC susceptibility with temperature is more complicated.

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

Method A—Standard Tensile Test

Method B—Standard Bent-Beam Test

Method C—Standard C-Ring Test

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

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

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

1.3.1 For testing at ambient conditions, the test procedures can be summarized as follows: Stressed test specimens are immersed in acidified aqueous environments containing H<sub>2</sub>S. Applied loads at convenient increments can be used to obtain EC data.

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

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

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

1.5 Safety Precautions: H<sub>2</sub>S is an extremely toxic gas that must be handled with care. (See Appendix A.)

## Section 2: EC Testing Variability

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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### Section 3: Reagents

#### 3.1 Reagent Purity

3.1.1 The test gases, sodium chloride (NaCl), acetic acid (CH<sub>3</sub>COOH), sodium acetate (CH<sub>3</sub>COONa), and solvents shall be reagent grade or chemically pure (99.5% minimum purity) chemicals. (See Appendix B.)

3.1.2 The test water shall be distilled or deionized and of quality equal to or greater than ASTM Type IV (ASTM D 1193<sup>6</sup>). Tap water shall not be used.

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

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### Section 4: Test Specimens and Material Properties

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

4.2 Tensile testing in accordance with standard test methods such as ASTM A 370<sup>7</sup> shall be used to determine base material properties. Two or more test specimens shall be pulled, and the individual test results shall be averaged to determine the yield and ultimate strengths, percent elongation, and percent reduction in area for the material. Machining a tensile test specimen from material adjacent to and in the same position and orientation as the EC test

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

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

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

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## Section 5: Test Vessels and Fixtures

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

5.2 Test vessels shall be capable of being purged to remove oxygen before beginning the test and of keeping air out during the test. Using a small outlet trap on the H<sub>2</sub>S effluent line to maintain 250 Pa (1.0 in H<sub>2</sub>O) back pressure on the test vessel prevents oxygen entry through small leaks or by diffusion up the vent line. (See Appendix B, section titled "Reasons for Exclusion of Oxygen.")

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

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

5.5 Test specimens shall be electrically isolated from test

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

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

### 5.7 Galvanic Coupling

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

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

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

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## Section 6: Test Solutions

### 6.1 Test Solution A

6.1.1 Test Solution A is an acidified and buffered, H<sub>2</sub>S-saturated aqueous brine solution.

6.1.2 Test Solution A shall consist of 5.0 wt% sodium chloride and 0.5 wt% glacial acetic acid dissolved in distilled or deionized water (e.g., 50.0 g of NaCl and 5.0 g of CH<sub>3</sub>COOH dissolved in 945 g of distilled or deionized water).

6.1.3 Test solution pH before or after H<sub>2</sub>S saturation but before contact with a test specimen is expected to range between 2.6 and 2.8. Each laboratory shall have a demonstrated and documented procedure for purging to verify that the pH has not exceeded 3.0 for Test Solution A after purging. During the test, pH may increase but shall not exceed 4.0. If the pH exceeds 4.0, the test is invalid. If the test-solution-volume to test-specimen-surface-area ratio is maintained and steps are taken to exclude oxygen from the test vessel as specified in this standard, the pH will not exceed this value.

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

### 6.2 Test Solution B

6.2.1 Test Solution B is an acidified and buffered, H<sub>2</sub>S-saturated aqueous brine solution.

6.2.2 Test Solution B shall consist of 5.0 wt% sodium chloride, 2.5 wt% glacial acetic acid, and 0.41 wt% sodium acetate dissolved in distilled or deionized water (e.g., 50.0 g of NaCl, 25 g of CH<sub>3</sub>COOH, and 4.1 g of CH<sub>3</sub>COONa dissolved in 921 g of distilled or deionized water).

6.2.3 Test solution pH before or after H<sub>2</sub>S saturation but before contact with a test specimen is expected to range between 3.4 and 3.6. During the test, pH may increase but shall not exceed 4.0. If the pH exceeds 4.0, the test is invalid. If the test-solution-volume to test-specimen-surface-area ratio is maintained and steps are taken to exclude oxygen from the test vessel as specified in this standard, the pH will not exceed this value.

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

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

### 6.3 Test Solution C

6.3.1 Test Solution C is a buffered aqueous brine solution with a chloride content, H<sub>2</sub>S partial pressure, and pH specified by the user or purchaser to simulate the intended service environment.

6.3.2 Test Solution C shall consist of distilled or deionized water containing 0.4 g/L sodium acetate (5 mM CH<sub>3</sub>COONa) and chloride (added as NaCl) at the same concentration as the intended service environment.

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

6.3.4 Test gas shall consist of a mixture of H<sub>2</sub>S and carbon dioxide (CO<sub>2</sub>), with H<sub>2</sub>S content sufficient to produce the specified H<sub>2</sub>S partial pressure of the intended service environment. The test gas shall be continuously bubbled through the test solution. The gas bubbling rate shall be optimized to maintain saturation of the test solution.

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

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

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

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

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

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

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

## Section 7: Testing at Elevated Temperature/Pressure

7.1 The dominant cracking mechanisms for most classes of materials in the presence of H<sub>2</sub>S vary with temperature. Ferritic steels and ferritic and martensitic stainless steels crack primarily by a hydrogen (i.e., cathodic) mechanism and have maximum susceptibility near room temperature. For austenitic stainless steels, as temperature increases, cracking susceptibility increases due to the major contribution from anodic processes. Duplex stainless steels exhibit mixed behavior, with maximum susceptibility to cracking in a mid-range of temperatures. To facilitate testing in simulated service conditions or to predict worst-case conditions, and to facilitate testing with H<sub>2</sub>S partial pressure exceeding 100 kPa (absolute) (14.5 psia), the following modified techniques are available.

7.2 Testing at elevated temperatures and pressures involves additional safety considerations compared with room temperature and atmospheric pressure testing. While some general guidance is given herein, it may not address all aspects and should be supplemented to accord with local safety requirements. Because H<sub>2</sub>S may be consumed during the test, gas replenishment and continuous gas

bubbling techniques are described. The H<sub>2</sub>S loss rate and its effect on the corrosiveness of the test environment are functions of several factors, including the corrosion rate of the test material and the partial pressure of H<sub>2</sub>S in the test environment. Guidance is given on measures that experience has shown to be appropriate for maintaining the required H<sub>2</sub>S partial pressure, but in all cases it is necessary to demonstrate, by measuring H<sub>2</sub>S concentration in either the test solution or gas phase, that the required test conditions have been maintained. This information must be reported with the test data.

### 7.3 Test Equipment

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

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



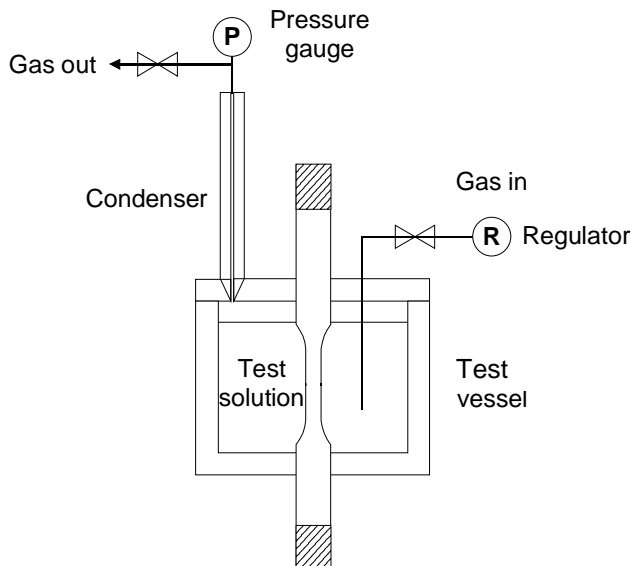
for gas, a dip tube on the inlet port, and a pressure-measuring device.

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

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

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

7.3.5 Elastomeric seal materials, if used, must resist  $\text{H}_2\text{S}$  at the temperature of use as verified by independent measurement.



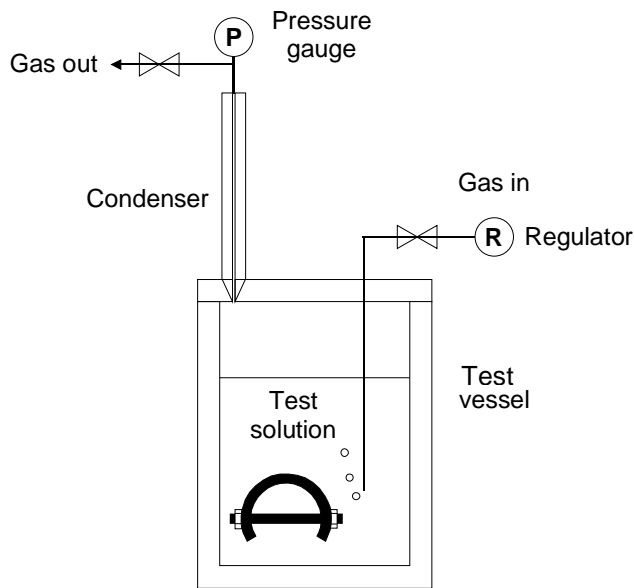
**FIGURE 1: Schematic Arrangement of Test Equipment for Method A—NACE Standard Tensile Test**

#### 7.4 Test Solution

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

#### 7.5 Test Gas

The test gas is usually a mixture of two or more of the following:  $\text{H}_2\text{S}$ ,  $\text{CO}_2$ , and inert gas such as  $\text{N}_2$  or Ar. At low  $\text{H}_2\text{S}$  partial pressures, tests in inert gas without  $\text{CO}_2$  require careful interpretation because of corrosion product solubility effects.<sup>11</sup> The test gas mixture should be contained in a standard gas bottle equipped with a suitable pressure regulator (usually stainless steel) capable of gas delivery to the total test pressure required. A commercially supplied gas mixture with composition determined by analysis should be used.



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

## 7.6 Test Procedure

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

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

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

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

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

7.6.4.1 Test vessel heated before test gas admitted

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

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

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

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

where:

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

7.6.4.2 Test gas admitted before test vessel heated

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

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

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

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

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

## Section 8: Method A—NACE Standard Tensile Test

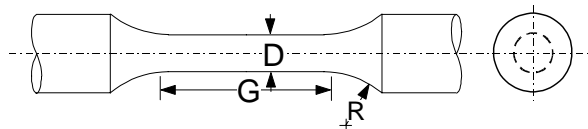
8.1 Method A, the NACE Standard Tensile Test, provides for evaluating metals for EC resistance under uniaxial tensile loading. It offers a simple unnotched test specimen with a well-defined stress state. EC susceptibility with Method A is usually determined by time-to-failure. Tensile test specimens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

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

### 8.2 Test Specimen

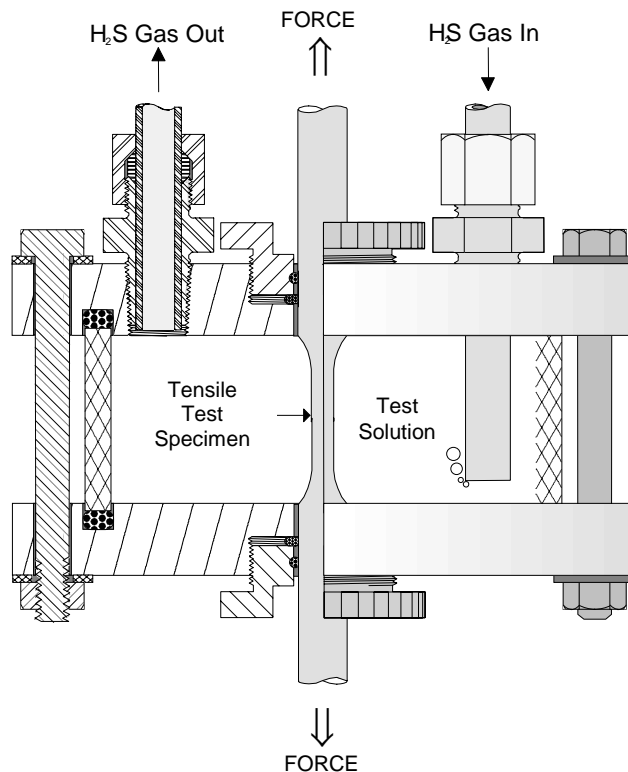
8.2.1 The size and shape of the material available for testing often restricts selection of test specimens. The orientation of the test specimen can affect the results and should be noted.

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



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

(a) Dimensions of the tensile test specimens



(b) Tensile test specimen in an environmental chamber

FIGURE 3: Tensile Test Specimens

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

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

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

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

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

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

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

#### 8.2.8 Test Specimen Identification

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

### 8.2.9 Test Specimen Cleaning

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

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

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

### 8.4 Test Equipment

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

8.4.2 Tensile tests should be performed with constant-load devices or sustained-load (proof ring or spring-loaded) devices (see ASTM G 49<sup>13</sup>).

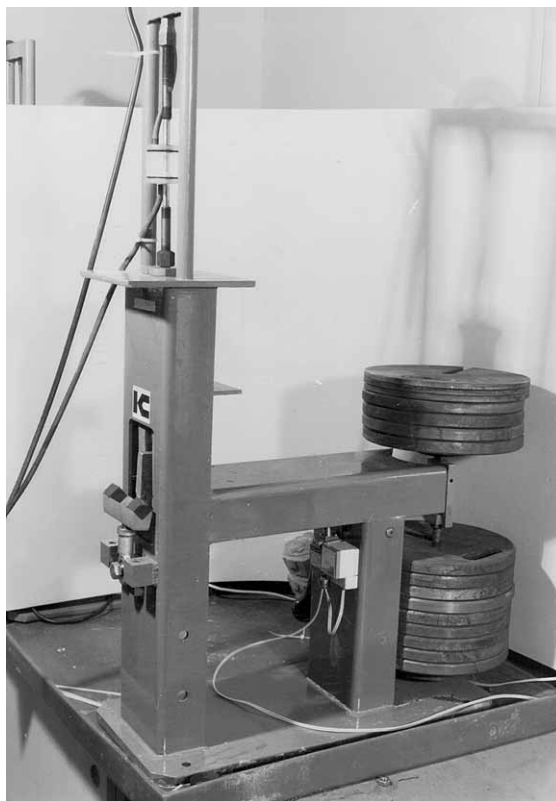
8.4.2.1 All loading devices shall be calibrated to ensure accurate application of load to the test specimen. The error for loads within the

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

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

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

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



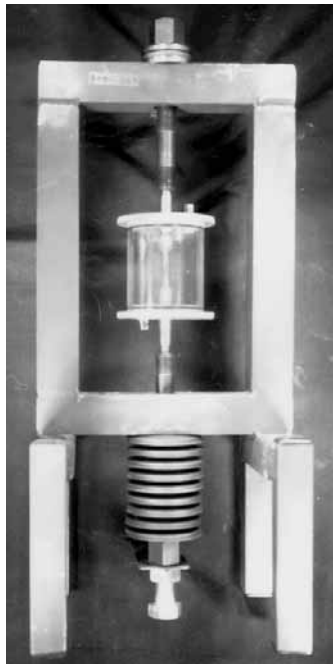
**FIGURE 4: Constant-Load (Dead-Weight) Device**

8.4.5 Sustained-load tests can be conducted with spring-loaded devices and proof rings when relaxation

in the fixtures or test specimen will result in only a small percentage decrease in the applied load (see Figure 5).



(a) Proof ring



(b) Spring-loaded

**FIGURE 5: Sustained-Load Devices**

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

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

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

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

- (a) the initiation and propagation of cracks in the test specimen;
- (b) yielding of the test specimen; or
- (c) relaxation of stress.

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

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

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

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

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

8.4.7 The test vessel shall be sized to maintain a test solution volume of  $30 \pm 10 \text{ mL/cm}^2$  of test specimen surface area.

## 8.5 Stress Calculations

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

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

where:

P = load;

S = applied stress; and

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

## 8.6 Testing Sequence

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

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

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

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

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

8.6.5 The test vessel shall immediately be filled with test solution such that the test specimen gauge section is fully immersed in the test solution. The test solution shall be completely deaerated by one of the following alternate methods to ensure that the test solution is oxygen-free before introducing  $\text{H}_2\text{S}$  (see Appendix B).

(a) The test solution may be deaerated within the test vessel by purging with inert gas at a rate of at least 100 mL/min for at least one hour.

(b) The test solution may be previously deaerated in a sealed vessel that is purged with inert gas at a rate of at least 100 mL/min for at least 1 h/L of test solution. After this previously deaerated test solution is transferred into the test vessel, it shall be purged with inert gas for at least 20 min after sealing the test vessel.

(c) Other methods of deaeration and transfer may be used if they result in a completely deaerated test solution before  $\text{H}_2\text{S}$  introduction.

8.6.6 The test solution shall then be saturated with  $\text{H}_2\text{S}$  at a rate of at least 100 mL/min for at least 20 min/L of test solution. A continuous flow of  $\text{H}_2\text{S}$  through the test vessel and outlet trap shall be maintained for the duration of the test at a low flow rate (a few bubbles per min). This maintains the  $\text{H}_2\text{S}$  concentration and a slight positive pressure to prevent air from entering the test vessel through small leaks.

8.6.6.1 Oxygen contamination is evident by a cloudy (opaque) appearance of the test solution when the  $\text{H}_2\text{S}$  gas enters the test vessel. An opaque appearance of the test solution upon  $\text{H}_2\text{S}$  entry shall disqualify the test. The test specimen shall be removed and cleaned, and the test



solution makeup, transfer, and deaeration procedure repeated.

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

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

## 8.7 Failure Detection

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

8.7.1 For all materials, failure is either:

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

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

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

## 8.8 Reporting of Test Results

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

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

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

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

9.1 Method B, the NACE Standard Bent-Beam Test, provides for testing carbon and low-alloy steels subjected to tensile stress to evaluate resistance to cracking failure in low-pH aqueous environments containing H<sub>2</sub>S. It evaluates EC susceptibility of these materials in the presence of a stress concentration. The compact size of the bent-beam test specimen facilitates testing small, localized areas and thin materials. Bent-beam test specimens loaded to a particular deflection give a failure/no-failure test result. When testing multiple test specimens at varying deflections, a statistically based critical stress factor ( $S_c$ ) for a 50% probability of failure can be obtained. NaCl is not added to the test solution for this test method. Laboratory test data for carbon and low-alloy steels have been found to correlate with field data.<sup>14</sup>

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

9.1.2 Method B can be summarized as follows:

9.1.2.1 This method involves deflecting each test specimen in a series by applying a different bending stress. The stressed test specimens then

are exposed to the test environment, and the failure (or no-failure) by cracking is determined. From these data obtained by testing multiple test specimens at varying deflections, a statistically based  $S_c$  for a 50% probability of failure is calculated to indicate the material's resistance to SSC.

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

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

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

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

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

**TABLE 1—NACE Uniform Material Testing Report Form (Part 1):  
Testing in Accordance with NACE Standard TM0177<sup>(A)</sup>  
Method A—NACE Standard Tensile Test**

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

	Heat Number/Identification					
<b>Chemistry</b>						
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) <sup>(B)</sup>						
Product Form						
Heat Treatment (Specify time, temperature, and cooling mode for each cycle in process.)						
Other Mechanical, Thermal, Chemical, or Coating Treatment <sup>(C)</sup>						

<sup>(A)</sup> Test method must be fully described if not in accordance with TM0177.

<sup>(B)</sup> Melt practice: open-hearth (OH), basic oxygen furnace (BOF), electric furnace (EF), argon-oxygen decarburization (AOD).

<sup>(C)</sup> E.g., cold work, plating, nitriding, prestrain.

**TABLE 1—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<sup>(A)</sup>

Test Specimen Geometry: ☐ Standard ☐ Nonstandard Nominal Diameter \_\_\_\_\_ Gauge Length \_\_\_\_\_  
 Constant Load—☐ Dead Weight ☐ Hydraulic ☐ Other \_\_\_\_\_  
 Sustained Load—☐ Proof Ring ☐ Spring ☐ Other \_\_\_\_\_  
☐ Post-Test Proof Ring Deflection Measurement

Chemistry: ☐ Test Solution A ☐ Test Solution B ☐ Test Solution C (define) \_\_\_\_\_ ☐ Other Test Solution \_\_\_\_\_

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

Material Identification	Test Specimen Properties							Test Stress (% of Yield Strength)								Test Solution pH <sup>(E)</sup>		Applied Heat Treatment	Remarks (Including Surface Condition and H <sub>2</sub> S Level)
	Location <sup>(B)</sup>	Orientation <sup>(C)</sup>	Yield Strength <sup>(D)</sup> ( )	Ultimate Tensile Strength ( )	Elongation (%)	Reduction in Area (%)	Hardness ( )								Start	Finish			
								Time-to-Failure (Hours) NF = No Failure at 720 hours											

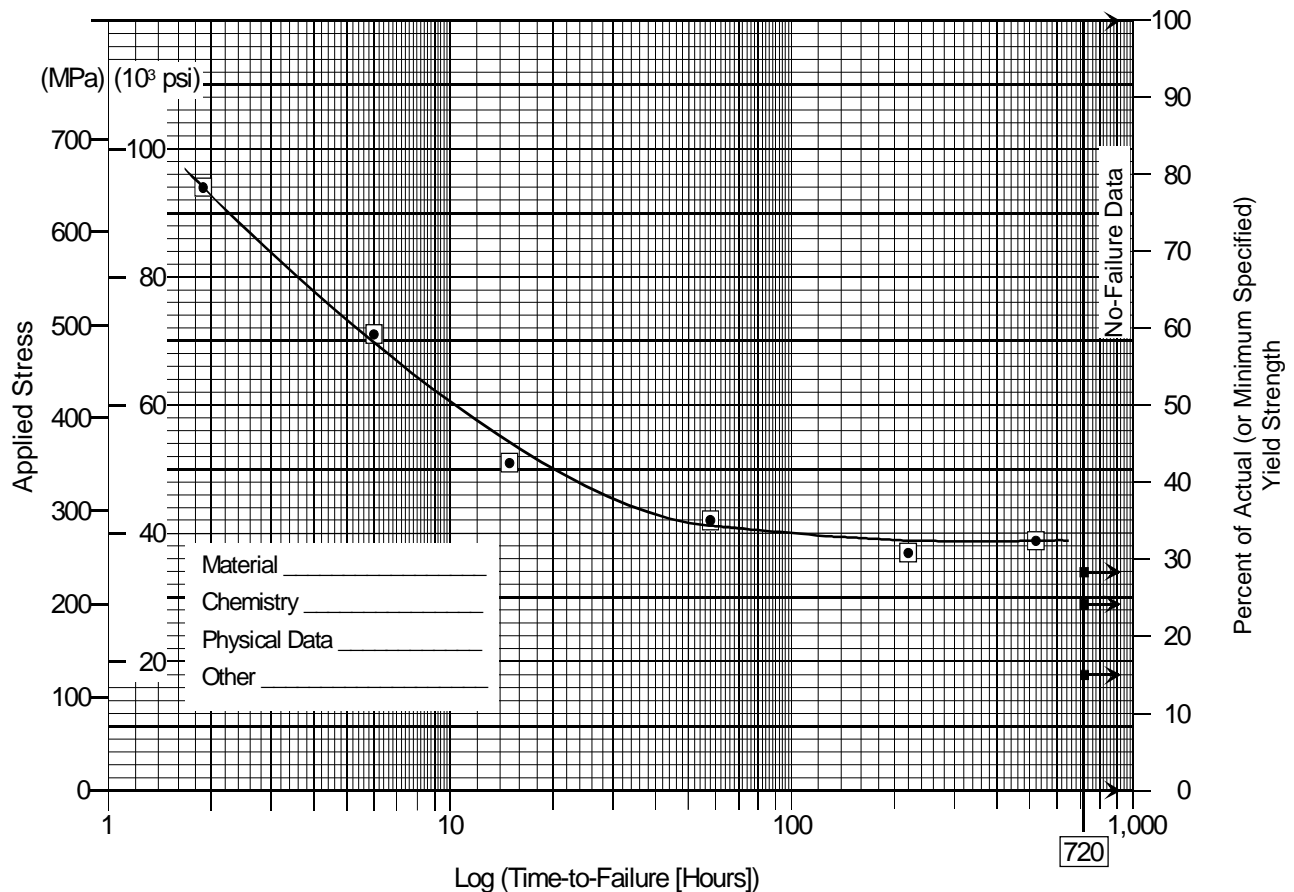
<sup>(A)</sup>Test method must be fully described if not in accordance with NACE Standard TM0177.

<sup>(B)</sup>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).

<sup>(C)</sup>Orientation may be longitudinal (L) or transverse (T).

<sup>(D)</sup>Open parentheses must be filled with metric or English units, as appropriate to the data. Yield strength is assumed to be 0.2% offset unless otherwise noted.

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



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

## 9.2 Test Specimen

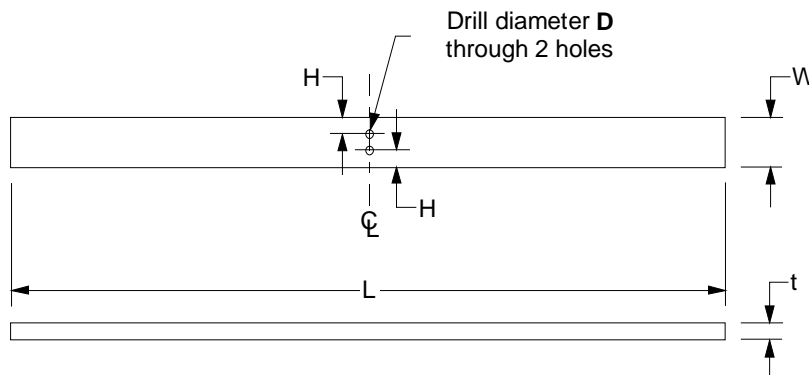
9.2.1 The dimensions of the standard bent-beam test specimen shall be  $4.57 \pm 0.13$  mm ( $0.180 \pm 0.0050$  in.) wide,  $1.52 \pm 0.13$  mm ( $0.060 \pm 0.0050$  in.) thick, and  $67.3 \pm 1.3$  mm ( $2.65 \pm 0.050$  in.) long (see Figure 7). After machining, test specimens shall be stored in a low-humidity area, in a desiccator, or in uninhibited oil until ready for testing.

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

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

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

9.2.4 As shown in Figure 7, two 0.71-mm (0.028-in.) diameter holes (No. 70 drill bit) must be drilled at the midlength of the test specimen, centered 1.58 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 ±0.01	0.028 ±0.0005 (No. 70 Drill)

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

#### 9.2.5 Test Specimen Identification

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

#### 9.2.6 Test Specimen Cleaning

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

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

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

#### 9.3 Test Solution

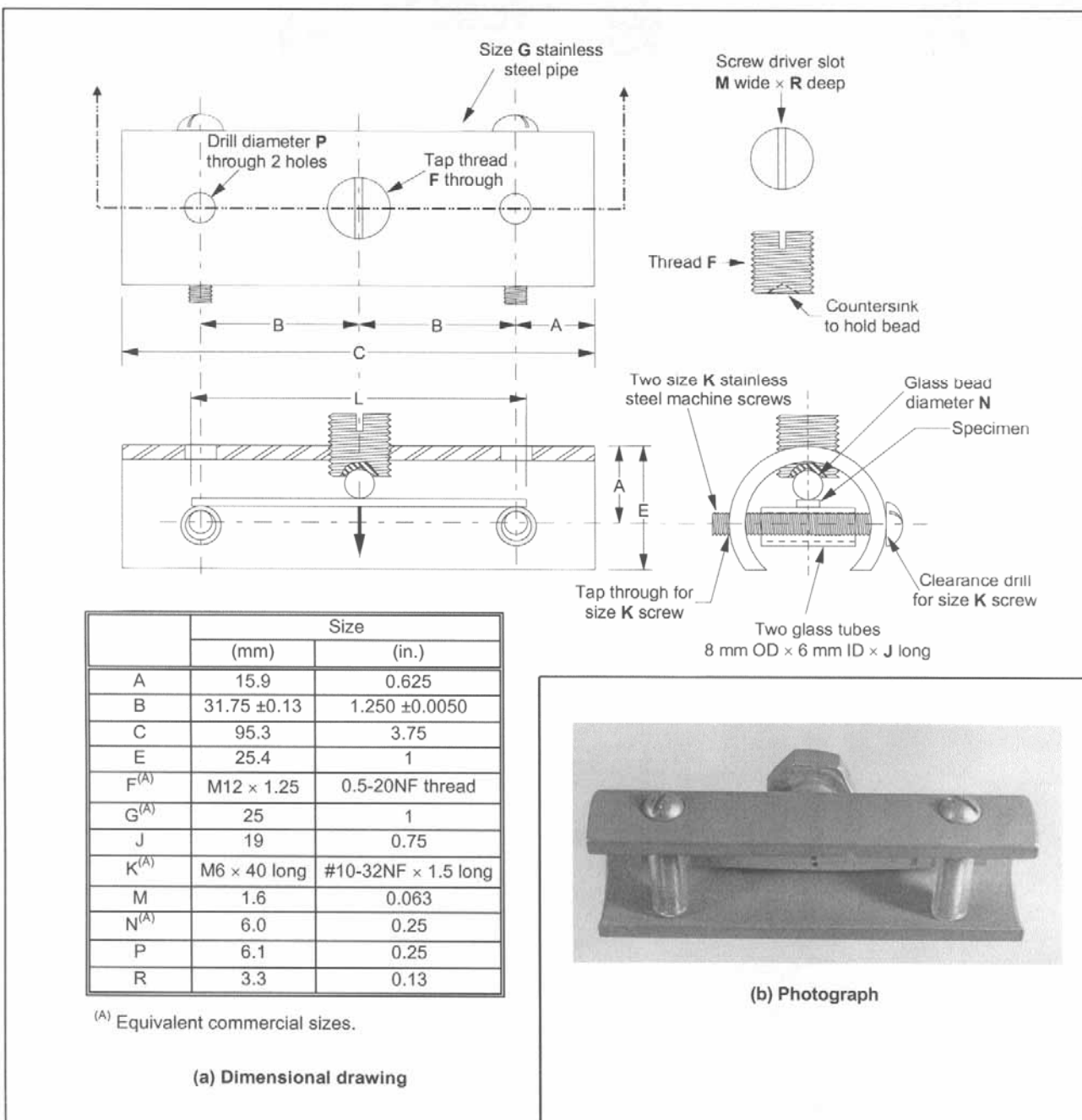
9.3.1 The test solution shall consist of 0.5 wt% glacial acetic acid dissolved in distilled or deionized water (e.g., 5.0 g of  $\text{CH}_3\text{COOH}$  dissolved in 995 g of distilled or deionized water). NaCl shall not be added to the test solution.

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

#### 9.4 Test Equipment

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

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



**FIGURE 8: Typical Stressing Fixture for Bent-Beam Test Specimen**

9.4.3 Test fixtures immersed in a test solution should resist general corrosion (UNS<sup>(4)</sup> S31600 is commonly used). Fixture elements contacting the test specimen must be electrically isolated from it.

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

<sup>(4)</sup> Metals and Alloys in the Unified Numbering System (latest revision), a joint publication of ASTM International and the Society of Automotive Engineers Inc. (SAE), 400 Commonwealth Dr., Warrendale, PA 15096.

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

#### 9.4.5 Test Vessel

9.4.5.1 The test vessel shall be sized to maintain a test solution volume of  $30 \pm 10 \text{ mL/cm}^2$  of test specimen surface area. Maximum volume of the test vessel should be 10 L.

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

9.4.5.3 A fritted glass bubbler shall be used to introduce the inert gas and  $\text{H}_2\text{S}$  below the array of test specimens. The bubbles should not impinge on the test specimens.

#### 9.5 Deflection Calculations

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

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

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

$$D = \frac{S\ell^2}{6Et} \quad (3)$$

where:

$D$  = deflection;

$S$  = nominal outer fiber pseudo-stress;

$\ell$  = distance between 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 dial gauge and fixture.

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

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

9.6.4 The test vessel shall be filled immediately with deaerated test solution and sealed. The test solution shall be completely deaerated by one of the following alternate methods to ensure that the test solution is oxygen-free before introducing  $\text{H}_2\text{S}$  (see Appendix B).

(a) The test solution may be deaerated within the test vessel by purging with inert gas at a rate of at least 100 mL/min for at least one hour.

(b) The test solution may be previously deaerated in a sealed vessel that is purged with inert gas at a rate of at least 100 mL/min for at least 1 h/L of test solution. After this previously deaerated test solution is transferred into the test vessel, it shall be purged with inert gas for at least 20 min after sealing the test vessel.

(c) Other methods of deaeration and transfer may be used if they result in a completely deaerated test solution prior to  $\text{H}_2\text{S}$  introduction.

9.6.5 The test solution shall then be saturated with  $\text{H}_2\text{S}$  at a rate of at least 100 mL/min for at least 20 min/L of test solution. The  $\text{H}_2\text{S}$  in the test vessel shall be replenished periodically by bubbling  $\text{H}_2\text{S}$  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 each week.

9.6.5.1 Oxygen contamination is evident by a cloudy (opaque) appearance in the test solution when the  $\text{H}_2\text{S}$  gas enters the test vessel. An opaque appearance to the test solution upon  $\text{H}_2\text{S}$  entry shall disqualify the test. The test specimen shall be removed and cleaned, and the test solution makeup, transfer, and deaeration procedures repeated.

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

9.6.7 Additional test specimens and iterative testing may be necessary to define the  $S_c$  closely.

## 9.8 Failure Detection

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

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

## 9.9 Reporting of Test Results

9.9.1 Failure/no-failure data and nominal outer fiber pseudo-stress ( $S$ ) values shall be reported. Time-to-failure data are optional.

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

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

where:

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

$T$  = the test result (i.e., +1 for passing and -1 for failure); and

$n$  = the total number of test specimens tested.

When using Equation (4), all pseudo-stress data that are more than  $\pm 210$  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.

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

where:

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

$T$  = the test result (i.e., +1 for passing and -1 for failure); and

$n$  = the total number of test specimens tested.

When using Equation (5), all pseudo-stress data that are more than  $\pm 3.0 \times 10^4$  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.

9.9.3 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.

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

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

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

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

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

### 10.2 Test Specimen

10.2.1 An unnotched C-ring test specimen in accordance with ASTM G 38<sup>15</sup> shall be used. Sizes for C-rings may be varied over a wide range, but C-rings with an outside diameter (OD) of less than about 15.9 mm (0.625 in.) should not be used because of



increased difficulties in machining and decreased precision in stressing. A typical C-ring test specimen is shown in Figure 9.

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

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

10.2.4 Machining should be done in stages: the final two passes should remove a total of no more than 0.05 mm (0.002 in.) of metal, and the final cut should leave the principal surface with a finish of 0.81  $\mu\text{m}$  (32  $\mu\text{in.}$ ) or finer. After machining, test specimens shall be stored in a low-humidity area, in a desiccator, or in uninhibited oil until ready for testing.

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

**TABLE 2—NACE Uniform Material Testing Report Form (Part 1):  
Testing in Accordance with NACE Standard TM0177<sup>(A)</sup>  
Method B—NACE Standard Bent-Beam Test**

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

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

<sup>(A)</sup> Test method must be fully described if not in accordance with TM0177.

<sup>(B)</sup> Melt practice: open-hearth (OH), basic oxygen furnace (BOF), electric furnace (EF), argon-oxygen decarburization (AOD).

<sup>(C)</sup> E.g., cold work, plating, nitriding, prestrain.

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

Lab Data for Material: \_\_\_\_\_ Tested per NACE Standard TM0177<sup>(A)</sup>

Test Specimen Geometry: ☐ Standard ☐ Nonstandard \_\_\_\_\_ Nominal Size \_\_\_\_\_ Length \_\_\_\_\_  
☐ Statistical S<sub>c</sub> Method Applied \_\_\_\_\_

Chemistry: ☐ 0.5 wt% glacial acetic acid in distilled or deionized water ☐ Other Test Solution \_\_\_\_\_

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

Material Identification	Test Specimen Properties							Psuedo-Stress (S) Value (      )								S <sub>c</sub> Value	Test Solution pH <sup>(E)</sup>		Applied Heat Treatment	Remarks (Including Surface Condition and H <sub>2</sub> S Level)
	Location <sup>(B)</sup>	Orientation <sup>(C)</sup>	Yield Strength <sup>(D)</sup> (      )	Ultimate Tensile Strength (      )	Elongation (%)	Reduction in Area (%)	Hardness (      )								Start		Finish			
								Time-to-Failure (Hours) NF = No Failure at 720 hours												

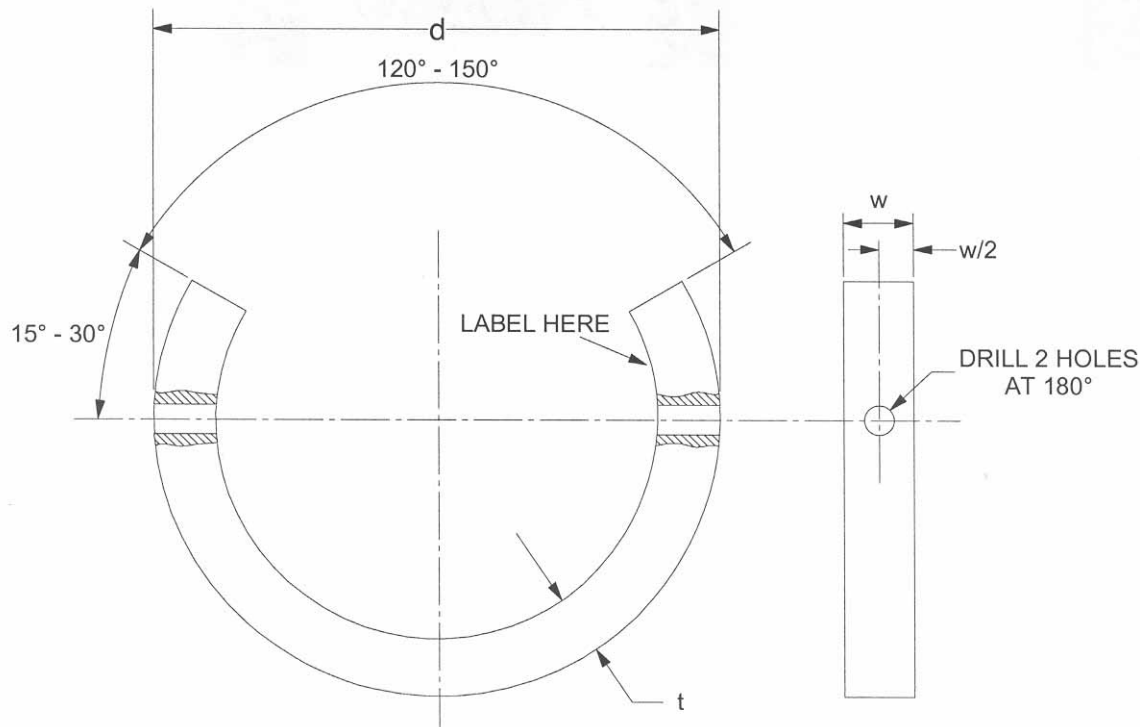
<sup>(A)</sup> Test method must be fully described if not in accordance with NACE Standard TM0177.

<sup>(B)</sup> 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).

<sup>(C)</sup> Orientation may be longitudinal (L) or transverse (T).

<sup>(D)</sup> Open parentheses must be filled with metric or English units, as appropriate to the data. Yield strength is assumed to be 0.2% offset unless otherwise noted.

<sup>(E)</sup> Enter pH for test conducted on nonfailed bent-beam test specimen at highest stress if summarizing data.



**FIGURE 9: Dimensional Drawing of the C-Ring Test Specimen**

#### 10.2.5 Test Specimen Identification

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

#### 10.2.6 Test Specimen Cleaning

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

10.2.6.2 After cleaning, the test section of the C-ring test specimen shall not be handled or contaminated.

#### 10.3 Test Solutions for Method C—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.

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

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

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

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

#### 10.4.3 Test Vessel

10.4.3.1 The test vessel should be sized to maintain a test solution volume of  $30 \pm 10 \text{ mL/cm}^2$  of test specimen surface area.

10.4.3.2 A fritted glass bubbler shall be used to introduce the inert gas and  $\text{H}_2\text{S}$  below the array of C-ring test specimens. The bubbles should not impinge on the C-ring test specimens.

#### 10.5 Deflection Calculations

10.5.1 The deflection necessary to obtain the desired stress on the C-ring test specimen shall be calculated using Equation (6):

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

where:

D = deflection of C-ring test specimen across bolt holes;

d = C-ring test specimen outer diameter;

t = C-ring test specimen thickness;

S = desired outer fiber stress; and

E = modulus of elasticity.

10.5.1.1 Deflections calculated by Equation (6) should be limited to stresses below the material elastic limit. For many CRAs the elastic limit is well below the 0.2% offset proof (yield) stress. Deflection values beyond the elastic limit can be calculated from information obtained from the stress-strain curve of the material and the strain-deflection characteristics of the specific C-ring geometry being used.

10.5.1.2 Equation (6) can be used to calculate the deflection necessary to stress the test specimen to 100% of the 0.2% offset yield strength ( $S_Y$ ) by substituting  $S_Y + E (0.002)$  for S in the original equation. This relationship is not valid for all alloy systems and should be checked before use on materials other than carbon and low-alloy steels.

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

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

10.5.2.1 Each C-ring shall be strain-gauged on the outside diameter at a point 90° opposite the axis of the C-ring bolt. The bolt shall be tightened to the appropriate strain by monitoring the strain gauge output, then the strain gauge and glue residue shall be removed. The C-ring shall then be recleaned using the same procedures given in Paragraph 10.2.6.

## 10.6 Testing Sequence

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

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

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

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

10.6.4 The test vessel shall be filled immediately with test solution and sealed. The test solution shall be completely deaerated by one of the following alternate methods to ensure that the test solution is oxygen-free before introducing  $H_2S$  (see Appendix B).

(a) The test solution may be deaerated within the test vessel by purging with inert gas at a rate of at least 100 mL/min for at least one hour.

(b) The test solution may be previously deaerated in a sealed vessel that is purged with inert gas at a rate of at least 100 mL/min for at least 1 h/L of test solution. After this previously deaerated test solution is transferred into the test vessel, it shall be purged with inert gas for at least 20 min after sealing the test vessel.

(c) Other methods of deaeration and transfer may be used if they result in a completely deaerated test solution prior to  $H_2S$  introduction.

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

10.6.5.1 Oxygen contamination is evident by a cloudy (opaque) appearance in the test solution when the  $H_2S$  gas enters the test vessel. An opaque appearance to the test solution upon  $H_2S$  entry shall disqualify the test. The test specimen shall be removed and cleaned, and the test solution makeup, transfer, and deaeration procedures repeated.

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

## 10.7 Failure Detection

10.7.1 Highly stressed C-rings of alloys that are appreciably susceptible to EC tend to fracture through the entire thickness or to crack in a way that is conspicuous. However, with more-EC-resistant alloys,

cracking frequently begins slowly and is difficult to detect. Small cracks may initiate at multiple sites and be obscured by corrosion products. It is preferable to report the first crack, if detected at 10X magnification, as the criterion of failure. An alternative method of exposing cracking in C-rings after exposure is to stress the C-ring beyond the tested stress level. Cracks resulting from EC can be differentiated from mechanically induced cracks by the corroded nature of the crack surface.

## 10.8 Reporting of Results

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

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

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

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## Section 11: Method D—NACE Standard DCB Test

11.1 Method D, the NACE Standard DCB Test, provides for measuring the resistance of metallic materials to propagation of EC, expressed in terms of a critical stress intensity factor,  $K_{ISSC}$  for SSC and  $K_{IEC}$  for the more general case of EC, using a crack-arrest type of fracture mechanics test. Method D does not depend on the uncertainty of pitting and/or crack initiation, because a crack is always initiated in a valid test. For SSC testing of carbon and low-alloy steels this method requires little time. Method D gives a direct numerical rating of crack propagation resistance and does not depend on evaluation of failure/no-failure results.<sup>16</sup> The subject of fracture mechanics testing for evaluation of EC resistance is currently under consideration by NACE TG 085 and Work Group (WG) 085c, and ASTM Committees E 8.06.02 and G 1.06.04. The user of this standard should maintain contact with these groups and their technical activities for knowledge of current state-of-the-art testing techniques.

11.1.1 This section sets forth the procedure for DCB testing at room temperature and atmospheric pressure

and enables computation of  $K_{ISSC}$ . When the special considerations set forth in Section 7 for testing at elevated temperature and pressure are observed, the computed stress intensity factor should be written as  $K_{IEC}$ . The equations needed to compute  $K_{IEC}$  are the same as those set forth in Paragraph 11.6 for  $K_{ISSC}$ . However, the following descriptions of material behavior under SSC conditions may not be accurate for the more general conditions of EC.

## 11.2 Test Specimen

11.2.1 The standard DCB test specimen design shall be in accordance with Figure 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. Wedges may be shielded with polytetrafluoroethylene (PTFE) tape to reduce corrosion in the wedge region.

**TABLE 3—NACE Uniform Material Testing Report Form (Part 1):  
Testing in Accordance with NACE Standard TM0177<sup>(A)</sup>  
Method C—NACE Standard C-Ring Test**

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

	Heat Number/Identification					
<b>Chemistry</b>						
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) <sup>(B)</sup>						
Product Form						
Heat Treatment (Specify time, temperature, and cooling mode for each cycle in process.)						
Other Mechanical, Thermal, Chemical, or Coating Treatment <sup>(C)</sup>						

<sup>(A)</sup> Test method must be fully described if not in accordance with TM0177.

<sup>(B)</sup> Melt practice: open-hearth (OH), basic oxygen furnace (BOF), electric furnace (EF), argon-oxygen decarburization (AOD).

<sup>(C)</sup> E.g., cold work, plating, nitriding, prestrain.

**TABLE 3—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<sup>(A)</sup>

Test Specimen Geometry: Outside Diameter \_\_\_\_\_ Wall/Thickness \_\_\_\_\_ Width \_\_\_\_\_

Test Equipment: ☐ Bolting Material Same as Specimen☐ Correction for Yield AppliedChemistry: ☐ Test Solution A ☐ Test Solution B ☐ Test Solution C (define) \_\_\_\_\_ ☐ Other Test Solution \_\_\_\_\_☐ Outlet Trap to Exclude Oxygen ☐ Temperature Maintained 24°C ±3°C (75°F ±5°F) ☐ Temperature Maintained \_\_\_\_\_ ±3°C (±5°F)

Material Identification	Test Specimen Properties							Applied Stress (% of Yield Strength)								Test Solution pH <sup>(E)</sup>		Applied Heat Treatment	Remarks (Including Surface Condition and H <sub>2</sub> S Level)
	Location <sup>(B)</sup>	Orientation <sup>(C)</sup>	Yield Strength <sup>(D)</sup> ( )	Ultimate Tensile Strength <sup>(D)</sup> ( )	Elongation (%)	Reduction In Area (%)	Hardness ( )								Start	Finish			
																	Time-to-Failure (Hours) NF = No Failure at 720 hours		

<sup>(A)</sup> Test method must be fully described if not in accordance with NACE Standard TM0177.<sup>(B)</sup> 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).<sup>(C)</sup> Orientation may be longitudinal (L) or transverse (T).<sup>(D)</sup> Open parentheses must be filled with metric or English units, as appropriate to the data. Yield strength is assumed to be 0.2% offset unless otherwise noted.<sup>(E)</sup> Enter pH for test conducted on nonfailed C-ring test specimen at highest stress if summarizing data.



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). When the material being tested is too thin to meet this requirement, optional thicknesses as noted in the figure may be considered. Subsize DCB test specimens of some carbon and low-alloy steels may give lower  $K_{ISSC}$  values than the standard DCB test specimens; differences as high as 37% have been observed. Further work is necessary to quantify this effect.

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

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

11.2.5 In testing materials of low  $K_{ISSC}$  (below 22 to 27  $\text{MPa}\sqrt{\text{m}}$  [20 to 25  $\text{ksi}\sqrt{\text{in.}}$ ]) or materials in which crack initiation is difficult, e.g., lower-yield-strength materials, introducing the electrodischarge-machined (EDM) slot noted in Figure 10(a) or a fatigue precrack is very helpful in avoiding side-cracking and in initiating SSC, respectively. Fatigue precracking of the DCB specimen should be conducted under load control at a convenient frequency. The acceptable range for a

fatigue precrack is 1 to 3 mm (0.04 to 0.12 in.) past the base of the chevron. To avoid erroneous high results, the maximum precracking load shall be the lesser of 70% of the expected initial  $K_I$  imparted by the wedge or 30  $\text{MPa}\sqrt{\text{m}}$  (27  $\text{ksi}\sqrt{\text{in.}}$ ). The ratio of minimum to maximum load shall be in the range of 0.1 to 0.2. The precrack should be sharpened at two-thirds of the maximum precracking load for approximately 20,000 cycles to extend the crack through any zone of residual compressive stresses that might have developed.

#### 11.2.6 Test Specimen Identification

11.2.6.1 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.

#### 11.2.7 Dimensional Check

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

#### 11.2.8 Test Specimen Cleaning

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

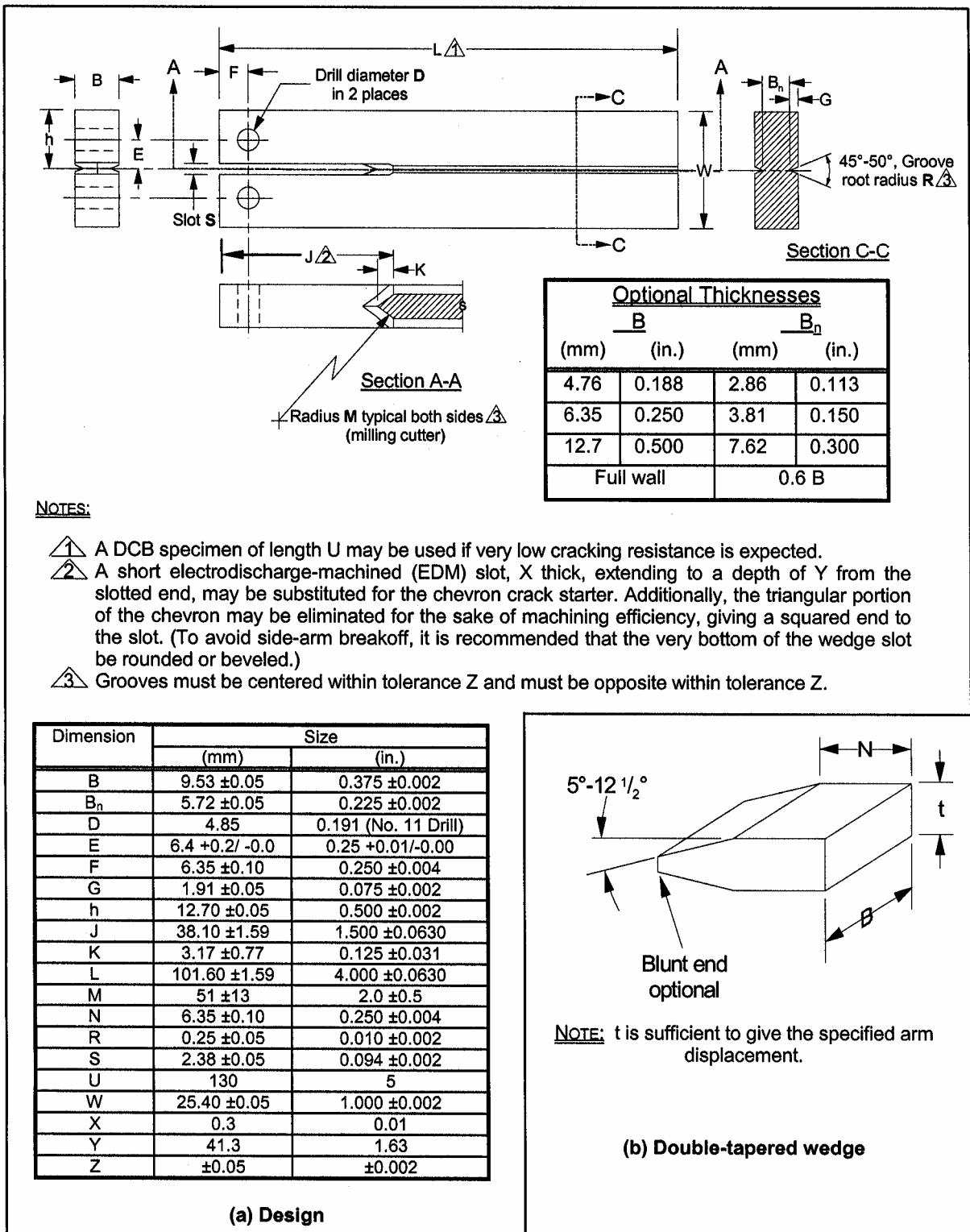


FIGURE 10: DCB Specimen

### 11.3 Test Solutions for Method D—see Section 6.

NOTE: For materials with high cracking resistance, fatigue precracking and/or higher initial arm displacements ( $\delta$ ) should be used with the higher-pH environment.

11.3.1 Subsequently, the test solution (Test Solution A, B, or C) shall be saturated with H<sub>2</sub>S as described in Paragraph 11.5.7.

### 11.4 Test Equipment

11.4.1 A test vessel sized to accommodate one liter of test solution per DCB test specimen (test solution volume of 10 to 12 mL/cm<sup>2</sup> of DCB test specimen surface area) should be employed. The maximum volume of the test vessel should be 10 L for ease of test solution saturation.

11.4.2 A slotted base plate or other test specimen holder (electrically isolating) should be used to ensure uniform spacing and orientation of the DCB test specimens.

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

11.4.4 A fritted glass bubbler shall be used to introduce the inert gas and H<sub>2</sub>S below the array of DCB test specimens. The bubbles should not impinge on the DCB test specimens.

### 11.5 Testing Sequence

11.5.1 Clean the DCB test specimens as indicated in Paragraph 11.2.8.1.

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

11.5.3 Measure the slot thickness. Feeler gauges should be used for this measurement. Because initial stress intensity affects the final K<sub>ISSC</sub> of carbon and low-alloy steels,<sup>17</sup> wedge thicknesses for the SSC-resistant grades of such steels that achieve the arm displacements in Table 4 should be selected. The arm displacement for each grade shall not exceed the upper limit listed in Table 4 for that grade.

The acceptable arm displacements given in Table 4 also may provide guidance in the selection of wedges for testing other grades of carbon and low-alloy steels. Table 5 provides guidance in choosing wedge thicknesses for low-alloy steel products not listed in Table 4 and for other alloys. When testing steels with yield strengths below 550 MPa (80 ksi), the arm displacement ( $\delta$ ) may be computed using Equation (7a) or (7b), in which YS is yield strength:

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

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

Recent experience has shown that carbon and low-alloy steels tested at elevated temperatures and/or milder test environments than Test Solution A may require greater arm displacements to achieve satisfactory crack initiation and/or growth than the arm displacements listed in Tables 4 and 5 for tests at room temperature. It is beyond the scope of this standard, however, to recommend specific displacement values for tests to be performed above room temperature or with milder test solutions. Preliminary experimentation therefore may be needed to establish appropriate arm displacements for such tests.

TABLE 4—Arm Displacements for API and Other Grade Oilfield Tubular Steels

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

<sup>(A)</sup> API grades unless noted otherwise.<sup>(B)</sup> Non-API grades.

TABLE 5—Suggested Arm Displacements for Selected Alloys and Strength Levels

Material		Arm Displacement ( $\delta$ ), mm (0.001 in.) <sup>(A)</sup>					
Yield Strength							
MPa	(ksi)	Low-Alloy Steels <sup>(B)</sup>		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)	—

<sup>(A)</sup> 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.<sup>(B)</sup> For oilfield tubular steels, use Table 4.

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

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

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

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

11.5.4 Degrease the DCB test specimens with solvent and rinse in acetone.

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

11.5.5 Place the DCB test specimens in the test vessel.

11.5.6 Add one liter of test solution per DCB test specimen (10 to 12 mL/cm<sup>2</sup> of DCB test specimen surface area) to the test vessel. The test solution shall be completely deaerated by one of the following alternate methods to ensure that the test solution is oxygen-free before introducing H<sub>2</sub>S (see Appendix B).

(a) The test solution may be deaerated within the test vessel by purging with inert gas at a rate of at least 100 mL/min for at least one hour.

(b) The test solution may be previously deaerated in a sealed vessel that is purged with inert gas at a rate of at least 100 mL/min for at least 1 h/L of test solution.

After this previously deaerated test solution is transferred into the test vessel, it shall be purged with inert gas for at least 20 min after sealing the test vessel.

(c) Other methods of deaeration and transfer may be used if they result in a completely deaerated test solution prior to H<sub>2</sub>S introduction.

11.5.7 Saturate the test solution with H<sub>2</sub>S at a rate of at least 100 mL/min for at least 20 min/L of test solution. A continuous H<sub>2</sub>S flow through the test vessel and outlet trap shall be maintained for the duration of the test at a low flow rate. This maintains the H<sub>2</sub>S concentration and a slight positive pressure to prevent air from entering the test vessel through small leaks.

11.5.7.1 Oxygen contamination is evident by a cloudy (opaque) appearance in the test solution when the H<sub>2</sub>S gas enters the test vessel. An opaque appearance to the test solution upon H<sub>2</sub>S entry shall disqualify the test. The test specimen shall be removed and cleaned, and the test solution makeup, transfer, and deaeration procedure repeated.

11.5.8 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<sub>ISSC</sub> values for low-alloy steels have been found to vary significantly with temperature in the vicinity of room temperature. Temperature of the test solution shall be monitored, and the average reported.

11.5.9 The test duration for carbon and low-alloy steels shall be 14 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. The duration of the test shall be mutually agreed upon among all interested parties and shall be reported with the results.

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

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

11.5.12 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<sub>ISSC</sub>

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

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

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

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

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

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

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

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

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

11.6.3 The stress intensity factor for SSC of flat DCB test specimens shall be calculated using Equation (8):

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

where:

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

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

#### 11.6.3.1 Calculation check:

Data:  $B = 9.53 \text{ mm}$  (0.375 in.);  
 $B_n = 5.72 \text{ mm}$  (0.225 in.);  
 $h = 12.7 \text{ mm}$  (0.500 in.);  
 $P = 1,870 \text{ N}$  (421 lb<sub>f</sub>);  
 $a = 46.48 \text{ mm}$  (1.830 in.); and  
 $K_{ISSC} = 35.3 \text{ MPa}\sqrt{\text{m}}$  (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 (9).<sup>18</sup>

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

where:

Equation (9); and

$K_{ISSC}$  (flat DCB test specimen) is calculated using

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

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

where:

Other data as in Paragraph 11.6.3.1.

$B$  and  $h$  have values in accordance with Paragraph 11.6.3; and

Result:

$D$  = tubular outside diameter.

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

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

#### 11.7 Reporting of Test Results:

##### 11.6.4.1 Calculation Check:

Data:

$D = 193.7 \text{ mm}$  (7.625 in.); and

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

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

11.7.3 Table 6 shows the recommended format for reporting the data.

**TABLE 6—NACE Uniform Material Testing Report Form (Part 1):  
Testing in Accordance with NACE Standard TM0177<sup>(A)</sup>  
Method D—NACE Standard DCB Test**

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

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

<sup>(A)</sup> Test method must be fully described if not in accordance with TM0177.

<sup>(B)</sup> Melt practice: open-hearth (OH), basic oxygen furnace (BOF), electric furnace (EF), argon-oxygen decarburization (AOD).

<sup>(C)</sup> E.g., cold work, plating, nitriding, prestrain.

**TABLE 6—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<sup>(A)</sup>

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

Chemistry: ☐ Test Solution A ☐ Test Solution B ☐ Test Solution C (define) \_\_\_\_\_ ☐ Other Test Solution \_\_\_\_\_

☐ Outlet Trap to Exclude Oxygen ☐ Temperature Maintained 24° ±1.7°C (75° ±3.0°F) ☐ Temperature Maintained \_\_\_\_\_ ±1.7°C (±3.0°F)

Material Identi- fication			DCB Test Specimen Properties <sup>(D)</sup>					Data for Valid Tests								Test Solution pH		Remarks (Including Surface Condition and H <sub>2</sub> S Level)
																Start	Finish	
	Location <sup>(B)</sup>	Orientation <sup>(C)</sup>	Yield Strength <sup>(D)</sup> ( )	Ultimate Tensile Strength ( )	Elongation (%)	Reduction in Area (%)	Hardness ( )	Arm Displacement (δ) ( )				K <sub>I</sub> <sub>SCC</sub> or K <sub>I</sub> <sub>EC</sub> ( )						
1								2	3	4	1	2	3	4	Mean			

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

<sup>(B)</sup> 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).

<sup>(C)</sup> Orientation may be longitudinal (L) or transverse (T).

<sup>(D)</sup> Open parentheses must be filled with metric or English units, as appropriate to the data. Yield strength is assumed to be 0.2% offset unless otherwise noted.<sup>(E)</sup> Enter pH for test conducted on nonfailed DCB test specimen at highest stress if summarizing data.



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19. OSHA<sup>(7)</sup> Rules and Regulations (Federal Register, Vol. 37, No. 202, Part II, October 18, 1972).
20. Chemical Safety Data Sheet SD-36 (Washington, DC: Manufacturing Chemists Association,<sup>(8)</sup> 1950).
21. N. Irving Sax, Dangerous Properties of Industrial Materials (New York, NY: Reinhold Book Corp., 1984).

<sup>(5)</sup> International Organization for Standardization (ISO), 1 rue de Varembe, Case Postale 56, CH-1121 Geneve 20, Switzerland.

<sup>(6)</sup> European Federation of Corrosion (Society of Chemical Industry), Institute of Metals, 1 Carlton House Terrace, London, SW1Y 5DB, UK.

<sup>(7)</sup> Occupational Safety and Health Administration (OSHA), 200 Constitution Ave. NW, Washington, DE 20210.

<sup>(8)</sup> Manufacturing Chemists Association (MCA), 1825 Connecticut Ave. NW, Washington, DC 20009.

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## Appendix A—Safety Considerations in Handling H<sub>2</sub>S Toxicity

H<sub>2</sub>S is perhaps responsible for more industrial poisoning accidents than is any other single chemical. A number of these accidents have been fatal. H<sub>2</sub>S must be handled with caution and any experiments using it must be planned carefully. The OSHA maximum allowable concentration of H<sub>2</sub>S in the air for an eight-hour work day is 20 parts per million (ppm), well above the level detectable by smell.<sup>19</sup> However, the olfactory nerves can become deadened to the odor after exposure of 2 to 15 min, depending on concentration, so that odor is not a completely reliable alarm system.

Briefly, the following are some of the human physiological reactions to various concentrations of H<sub>2</sub>S. Exposure to concentrations in the range of 150 to 200 ppm for prolonged periods may cause edema of the lungs. Nausea, stomach distress, belching, coughing, headache, dizziness, and blistering are symptoms of poisoning in this range of concentration. Pulmonary complications, such as pneumonia, are strong possibilities from such subacute exposure. At 500 ppm, unconsciousness may occur in less than 15 min, and death within 30 min. At concentrations above 1,000 ppm, a single inhalation may result in instantaneous unconsciousness, complete respiratory failure, cardiac arrest, and death.

Additional information on the toxicity of H<sub>2</sub>S can be obtained from the Chemical Safety Data Sheet SD-36<sup>20</sup> and from *Dangerous Properties of Industrial Materials*.<sup>21</sup>

### Fire and Explosion Hazards

H<sub>2</sub>S is a flammable gas and yields poisonous sulfur dioxide (SO<sub>2</sub>) as a combustion product. In addition, its explosive limits range from 4 to 46% in air. Appropriate precautions shall be taken to prevent these hazards from developing.

### Safety Procedures During Test

All tests shall be performed in a hood with adequate ventilation to exhaust all of the H<sub>2</sub>S. The H<sub>2</sub>S flow rates during the test should be kept low to minimize the quantity exhausted. A 10% caustic absorbent solution for effluent gas can be used to further minimize the quantity of H<sub>2</sub>S gas exhausted. This caustic solution needs periodic replenishing. Provision shall be made to prevent backflow of the caustic solution into the test vessel if the H<sub>2</sub>S flow is interrupted. Suitable safety equipment shall be used when working with H<sub>2</sub>S.

Because the downstream working pressure frequently rises as corrosion products, debris, etc., accumulate and interfere with regulation at low flow rates, particular attention should be given to the output pressure on the pressure regulators. Gas cylinders shall be securely fastened to prevent tipping and breaking of the cylinder head. Because H<sub>2</sub>S is in liquid form in the cylinders, the high-pressure gauge must be checked frequently, because relatively little time elapses after the last liquid evaporates and the pressure drops from 1.7 MPa (250 psig) to atmospheric pressure. The cylinder shall be replaced by the time it reaches 0.5 to 0.7 MPa (75 to 100 psig) because the regulator control may become erratic. Flow shall not be allowed to stop without closing a valve or disconnecting the tubing at the test vessel because the test solution continues to absorb H<sub>2</sub>S and move upstream into the flowline, regulator, and even the cylinder. A check valve in the line should prevent the problem if the valve works properly. However, if such an accident occurs, the remaining H<sub>2</sub>S should be vented as rapidly and safely as possible and the manufacturer notified so that the cylinder can be given special attention.

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## Appendix B—Explanatory Notes on EC Test Method

### Reasons for Reagent Purity

Water impurities of major concern are alkaline- or acid-buffering constituents that may alter the pH of the test solution and organic and inorganic compounds that could change the nature of the corrosion reaction. Oxidizing agents could also convert part of the H<sub>2</sub>S 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<sub>2</sub>S to obtain a lower partial pressure of H<sub>2</sub>S in the gas and hence a lower H<sub>2</sub>S concentration in the test solution. Oxidation products could accumulate, resulting in changes in corrosion rate and/or hydrogen entry rate (see the paragraph below on Reasons for Exclusion of Oxygen).

### Preparation of Test Specimen

All machining operations shall be performed carefully and slowly so that overheating, excessive gouging, and cold work do not alter critical physical properties of the material.

Uniform surface condition is critical to consistent SSC test results.

### Reasons for Exclusion of Oxygen

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

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

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

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

### Cautionary Notes

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