



ANSI/NACE Standard TM0284-2011
Item No. 21215

Standard Test Method

Evaluation of Pipeline and Pressure Vessel Steels for Resistance to Hydrogen-Induced Cracking

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Foreword

Absorption of hydrogen generated by corrosion of steel in a wet hydrogen sulfide (H_2S) environment can have several effects that depend on the properties of the steel, manufacturing or forming processes, the characteristics of the environment, and other variables. One adverse effect observed in pipeline and pressure vessel steels is the development of cracks along the rolling direction of the steel. Cracks on one plane tend to link up with the cracks on adjacent planes to form steps across the thickness. The cracks can reduce the effective wall thickness until the pipeline or pressure vessel becomes overstressed and ruptures. Cracking is sometimes accompanied by surface blistering. Several service failures attributed to such cracking have been reported.^{1,2}

The terms *stepwise cracking* (SWC), *hydrogen pressure cracking*, *blister cracking*, and *hydrogen-induced stepwise cracking* have been used in the past to describe cracking of this type in pipeline and pressure vessel steels, but are now considered obsolete. The term *hydrogen-induced cracking* (HIC) has been widely used for describing cracking of this type, and has been adopted by NACE International. Therefore, it is used throughout this standard test method.

HIC is related to hydrogen blistering, which has been recognized since the 1940s as a problem in pressure vessels handling sour products.³ It was not until much later, however, that HIC gained wide recognition as a potential problem in pipelines. As a result of pipeline failures experienced by two companies in the early 1970s, several companies began investigating the cracking and publishing results of tests on various steels. Many investigators found, however, that they could not reproduce published test results. It was eventually determined that lack of reproducibility resulted largely from differences in test procedures. Consequently, NACE Unit Committee T-1F, "Metallurgy of Oilfield Equipment," established Task Group (TG) T-1F-20 to study the problem and prepare a standard test method.

This standard was originally prepared in 1984 to provide a standard set of test conditions for consistent evaluation of steel pipes and for comparison of test results from different laboratories. Subsequently, the concern for HIC damage turned to steel plates used for pressure vessels. Requirements for testing steel plates for resistance to HIC were included in this standard in 1996. More recently, concern for HIC damage in steel fittings and flanges used in pipelines and pressure vessels has led to their inclusion in the 2011 revision of this standard. Therefore, the scope of this standard now includes the testing of steels furnished in the form of pipes, plates, fittings, and flanges for use in fabricating pipelines and pressure vessels.

Test conditions are not designed to simulate any particular pipeline or process operation. The test is intended to evaluate resistance to HIC only, and not other adverse effects of sour environments such as sulfide stress cracking (SSC), pitting, or mass loss from corrosion.

This test may be used for many purposes, and the applications of the results are beyond the scope of this standard. Those who use the test should be aware that in some cases test results can be influenced by variations in properties among different locations in a single length of pipe or individual plate, fitting, or flange, as well as by variations within a heat of steel. When the test is used as a basis for purchasing, the number and location of test specimens must be carefully considered.⁴ This standard is intended for end users, manufacturers, fabricators, and testing laboratories.

This standard was originally prepared by Task Group T-1F-20 in 1984. It was revised by TG T-1F-20 in 1996, and in 2003 and 2011 by Task Group (TG) 082, "Stepwise Cracking of Pipeline Steels." This standard is published by NACE under the auspices of Specific Technology Group (STG) 32, "Oil and Gas Production—Metallurgy." It 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*. The terms *shall* and *must* are used to state a requirement, and are considered mandatory. The term *should* is used to state something good and is recommended, but is not considered mandatory. The term *may* is used to state something considered optional.

NACE International Standard Test Method

Evaluation of Pipeline and Pressure Vessel Steels for Resistance to Hydrogen-Induced Cracking

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Section 1: General

1.1 This standard establishes a test method for evaluating the resistance of pipeline and pressure vessel steels to HIC caused by hydrogen absorption from aqueous sulfide corrosion.

1.1.1 Details are provided on the size, number, location, and orientation of test specimens to be taken from each steel product form—pipes, plates, fittings, and flanges.

1.1.2 Special procedures or requirements for testing small-diameter (nominal diameter [DN] 50 through 150, nominal pipe size [NPS] 2 through 6), thin-wall (up to 6 mm [0.2 in] wall thickness), electric-resistance welded (ERW) and seamless pipes are included. The test specimens taken from small-diameter, thin-wall pipes shall be tested in the same manner as the test specimens taken from other pipes except as otherwise stated in this standard.

1.2 The test method consists of exposing unstressed test specimens to one of the two standard test solutions—Test Solution A, a solution consisting of sodium chloride (NaCl) and acetic acid (CH₃COOH) dissolved in distilled or deionized water saturated with H₂S at ambient temperature and pressure; or Test Solution B, a synthetic seawater solution saturated with H₂S at ambient temperature and pressure. After a specified time the test specimens are removed and evaluated.

1.3 The test method is not intended to duplicate service conditions. It is intended to provide reproducible test environments capable of distinguishing the susceptibility of different steel samples to HIC in a relatively short time. NOTE: The length of the test may not be sufficient to develop maximum cracking in any given steel, but has been found to be adequate for the purpose of this test.

1.4 This standard does not include acceptance or rejection criteria; however, guidance is provided in NACE MR0175/ISO⁽¹⁾ 15156,⁵ Part 2, Section 8 and Annex B of EFC⁽²⁾ 16.⁶

Section 2: Reagents

2.1 The reagents for Test Solution A shall be nitrogen gas for purging, H₂S gas, NaCl, CH₃COOH, and distilled or deionized water. The reagents for Test Solution B shall be nitrogen gas for purging, H₂S gas, and synthetic seawater.

NOTE: H₂S is highly toxic and must be handled with caution. See Appendix A (nonmandatory).

2.2 The NaCl and CH₃COOH shall be reagent grade chemicals.

2.3 The gases shall be reagent grade or chemically pure gases and the water shall be distilled or deionized. See Appendix B (nonmandatory).

2.4 The synthetic seawater shall be prepared in accordance with ASTM⁽³⁾ Standard D1141,⁷ Stock Solutions No. 1 and No. 2 (without heavy metal ions).

⁽¹⁾ International Organization for Standardization (ISO), 1 ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland.

⁽²⁾ European Federation of Corrosion, 1 Carlton House Terrace, London, SW1Y 5DB, U.K.

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

Section 3: Testing Apparatus

3.1 Figure 1 is a schematic diagram of a typical test assembly.

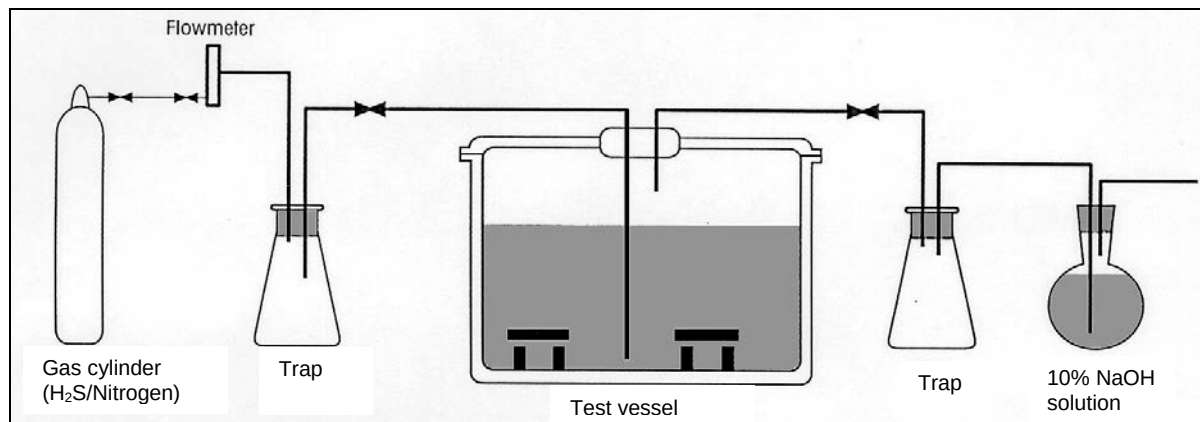


Figure 1: Schematic diagram of typical test assembly.

3.2 The test may be performed in any convenient airtight test vessel large enough to contain the test specimens with provisions for purging and introduction of H₂S.

3.3 None of the materials involved in the test set-up shall contaminate or react with the test environment.

Section 4: Test Specimens—Pipes

4.1 Size

4.1.1 Each test specimen shall be 100 ± 1 mm (4.00 ± 0.04 in) long by 20 ± 1 mm (0.80 ± 0.04 in) wide.

4.1.2 The test specimen thickness shall be the full wall thickness of the pipe up to a maximum of 30 mm (1.2 in). For wall thickness greater than 30 mm (1.2 in), the test specimen thickness shall be either the full wall thickness of the pipe or limited to a maximum thickness of 30 mm (1.2 in) and staggered through the thickness, as described in Section 5. A maximum of 1 mm (0.04 in) may be removed from each of the surfaces (i.e., internal and external). Test specimen blanks shall not be flattened.

4.1.3 For small-diameter, thin-wall ERW and seamless pipe, the test specimen thickness must be at least 80% of the full wall thickness of the pipe. In such cases, curved test specimens cut from the pipe shall be tested; test specimen blanks shall not be flattened.

4.2 Number, Location, and Orientation

4.2.1 Three test specimens shall be taken from each test pipe.

4.2.2 For seam-welded pipe, the test specimens shall be taken from the weld, 90 degrees from the weld, and 180 degrees from the weld. For seamless pipe, the test specimens shall be taken 120 degrees apart around the circumference.

4.2.3 Test specimens shall be taken from the pipe with the longitudinal axis of the test specimens:

- (a) parallel to the longitudinal axis of the pipe for seamless pipe and the parent metal of longitudinally welded pipe;
- (b) parallel to the weld for the parent metal of spiral-welded pipe;
- (c) perpendicular to the weld for the weld area of longitudinally and spiral-welded pipe; and

- (d) parallel to the weld for the weld area of ERW pipe. The weld shall be approximately on the center line of the test specimen.

Figures 2 through 6 show the orientation of test specimens and where they shall be sectioned and examined after exposure.

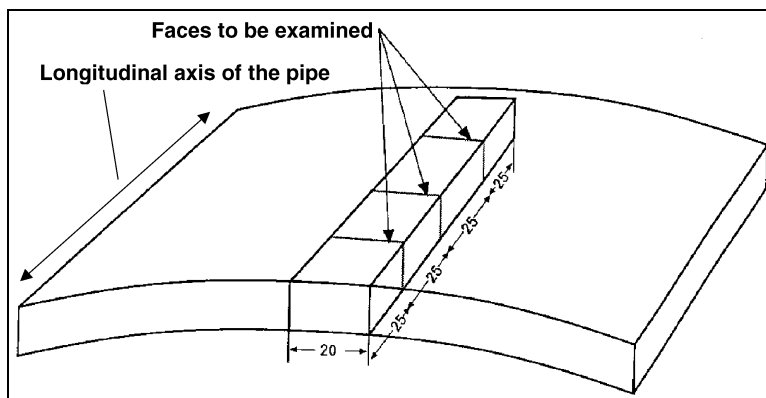


Figure 2: Seamless pipe and parent metal of longitudinally welded pipe (all dimensions in mm [1 in = 25.4 mm]).

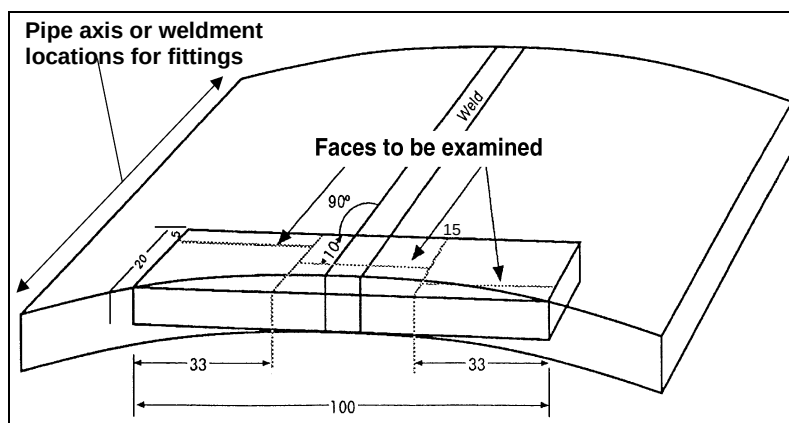


Figure 3: Weld area of longitudinally welded pipe or welded fittings (all dimensions in mm [1 in = 25.4 mm]).

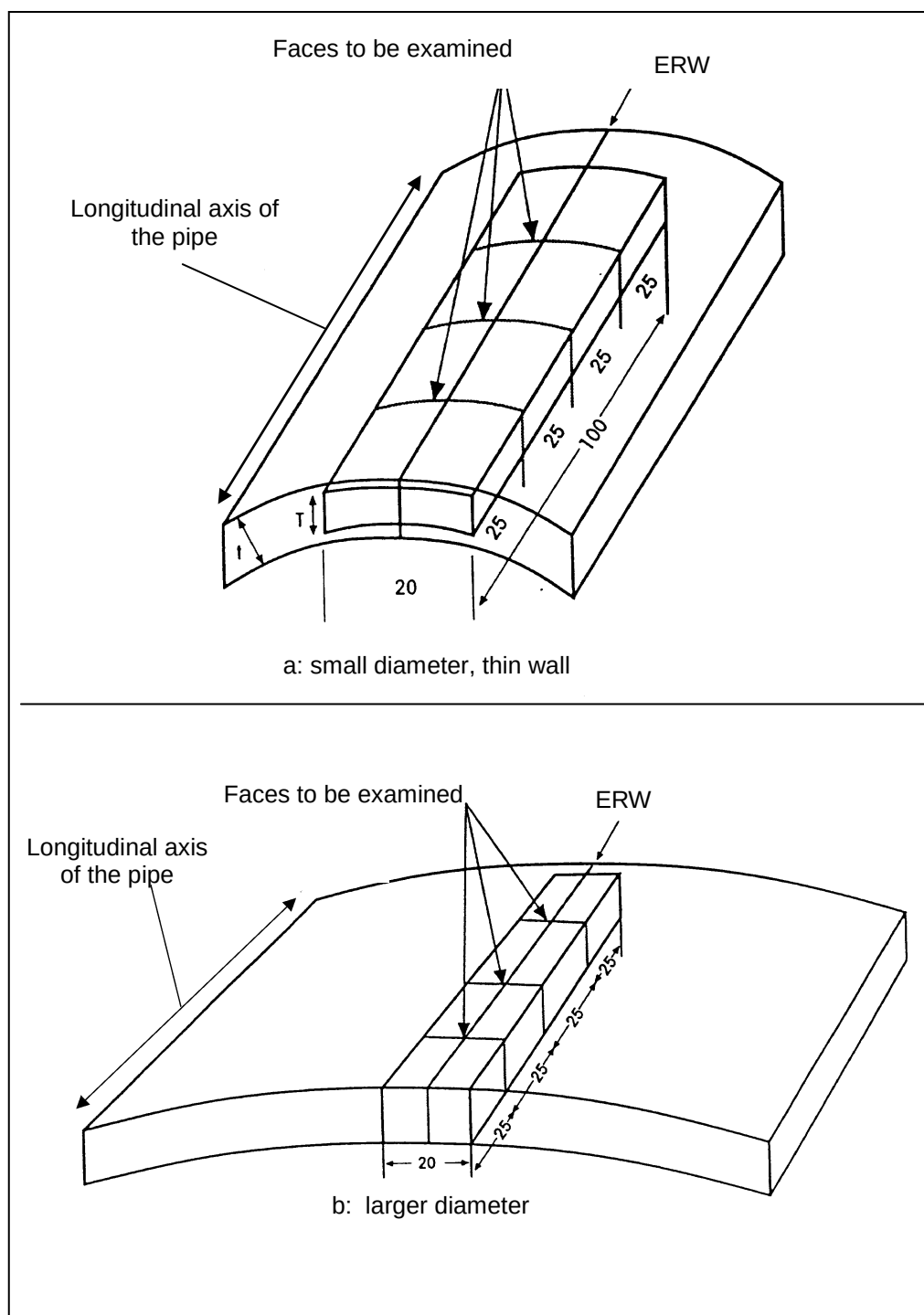


Figure 4: Weld area of ERW pipe (all dimensions in mm [1 in = 25.4 mm]).

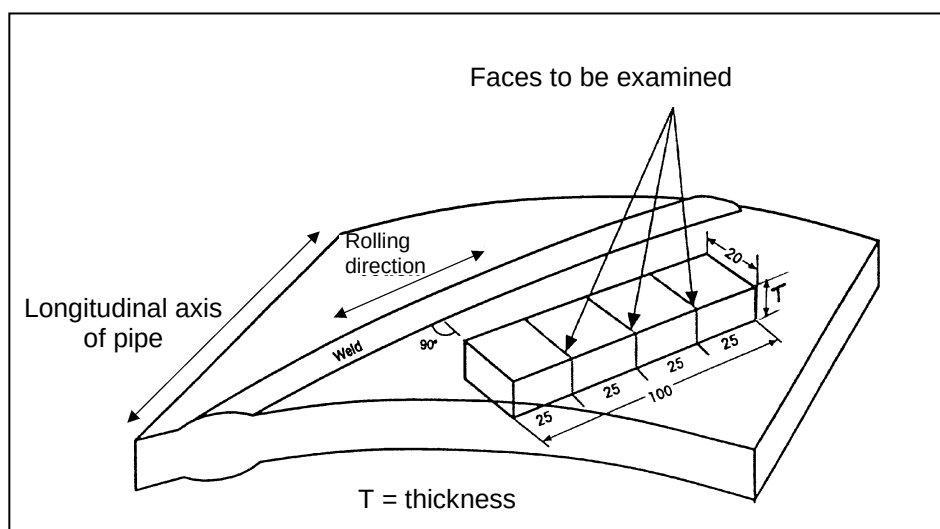


Figure 5 (a): Parent metal of spiral-welded pipe (all dimensions in mm [1 in = 25.4 mm]).

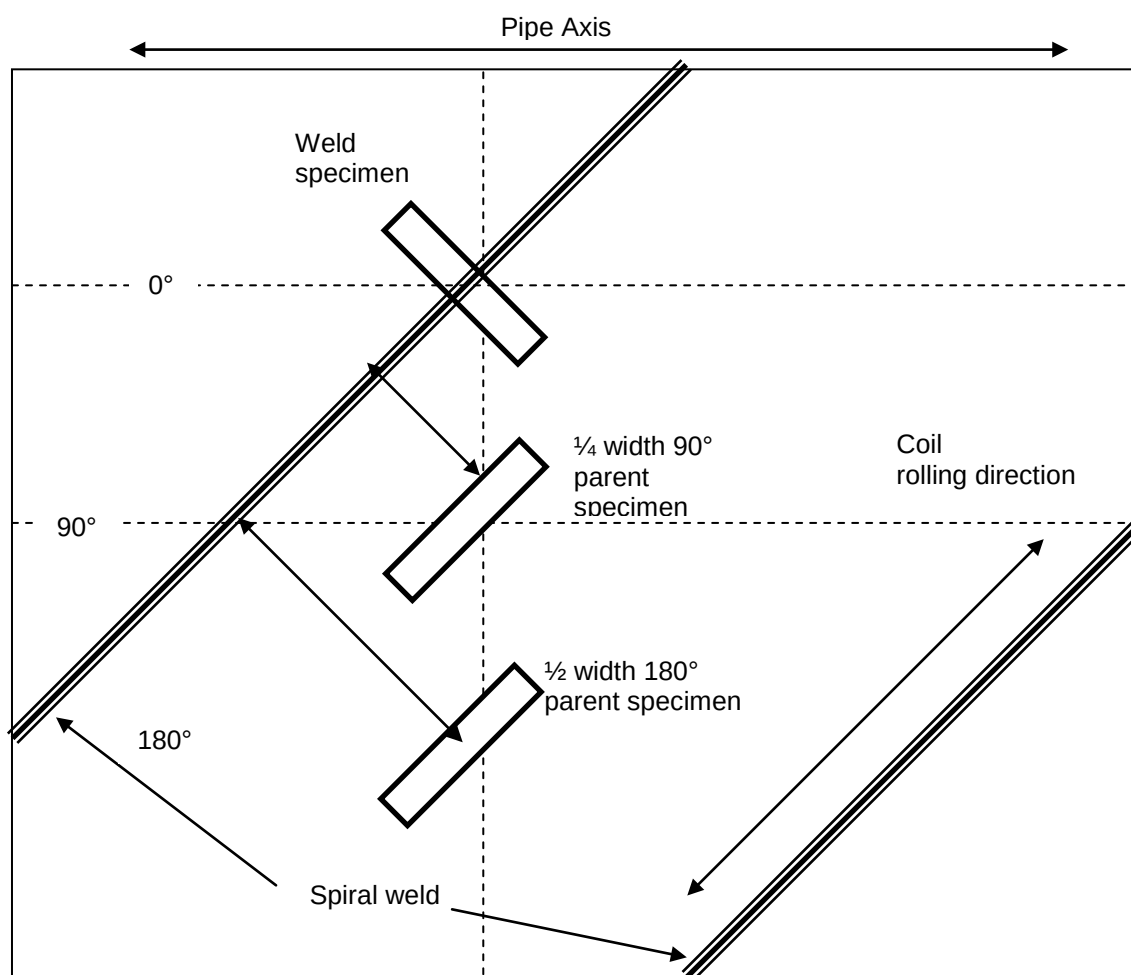


Figure 5 (b): Location and orientation of test specimens to be taken from spiral-welded pipe.

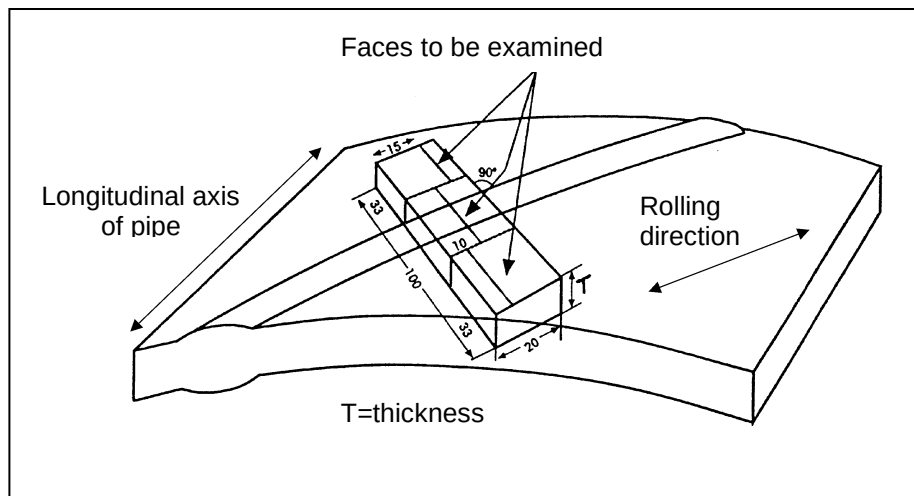


Figure 6: Weld area of spiral-welded pipe (all dimensions in mm [1 in = 25.4 mm]).

4.3 Preparation

4.3.1 Blanks for test specimens may be removed by any convenient method. If a blank is torch cut, the heat-affected zone of the torch-cut surface shall be completely removed by grinding, sawing, or machining.

4.3.2 The four cut edge surfaces of each test specimen shall be ground (wet or dry) and finished with 320 grit paper.

4.3.3 Coating of the cut edge surfaces is not allowed; all six surfaces shall be exposed to the test solution.

4.3.4 Small-diameter, thin-wall ERW and seamless pipe test specimens shall have all mill scale removed from the internal and external surfaces. Either machining and/or grinding (wet or dry) shall result in a bare metal surface with a finish equivalent to 320 grit. For machining, the last two passes shall be such that a maximum of 0.05 mm (0.002 in) of material is removed.

4.4 Cleaning and Storing

4.4.1 Prior to testing, the test specimens shall be degreased with a suitable degreasing solution and rinsed with an appropriate solvent, such as acetone. The adequacy of the degreasing method shall be determined for each batch of test specimens by the atomizer test in accordance with ASTM F21⁸ or another equivalent method. The method used shall be reported.

4.4.2 Test specimens may be stored in a desiccator after degreasing and shall be verified for the adequacy of degreasing using the ASTM F21 test or equivalent between removal from the desiccator and exposure to the test solution.

Section 5: Test Specimens—Plates

5.1 Size

5.1.1 Each test specimen shall be 100 ± 1 mm (4.00 ± 0.04 in) long by 20 ± 1 mm (0.80 ± 0.04 in) wide.

5.1.2 A maximum of 1 mm (0.04 in) may be removed from the rolled surfaces. Test specimen blanks shall not be flattened.

5.1.3 The test specimen thickness shall be the full thickness of the plate, up to a maximum of 30 mm (1.2 in). For plates thicker than 30 mm (1.2 in), the test specimens shall be staggered as indicated in Paragraphs 5.2.3 and 5.2.4, with the

following exception: for plates thicker than 30 mm (1.2 in) that are intended to be used for manufacture of pipe, the test specimens may be full wall thickness in accordance with Paragraph 4.1.2.

5.2 Number, Location, and Orientation

5.2.1 The test specimen location for plates shall be at one end, mid-width of the plate, with the longitudinal axis of the test specimen parallel to the principal rolling direction of the plate.

5.2.2 For plates up to 30 mm (1.2 in) thick, inclusive, three test specimens shall be taken as shown in Figure 7.

5.2.3 For plates over 30 mm (1.2 in) to 88 mm (3.5 in) thick, inclusive, three test specimens, each 30 mm (1.2 in) thick, located near both surfaces and at the center line shall be taken to provide for testing of the full plate thickness, as shown in Figure 8. The test specimens shall be evenly staggered in the through-thickness direction, with the overlap in the through-thickness direction being determined by the actual plate thickness. However, the minimum overlap shall be 1 mm (0.04 in) between adjacent test specimens.

5.2.4 For plates over 88 mm (3.5 in) thick, five or more test specimens (there must be an uneven number), each 30 mm (1.2 in) thick, shall be taken as shown in Figure 9. The test specimens shall be evenly staggered in the through-thickness direction, with the overlap in the through-thickness direction being determined by the actual plate thickness. The minimum overlap shall be 1 mm (0.04 in) between adjacent test specimens.

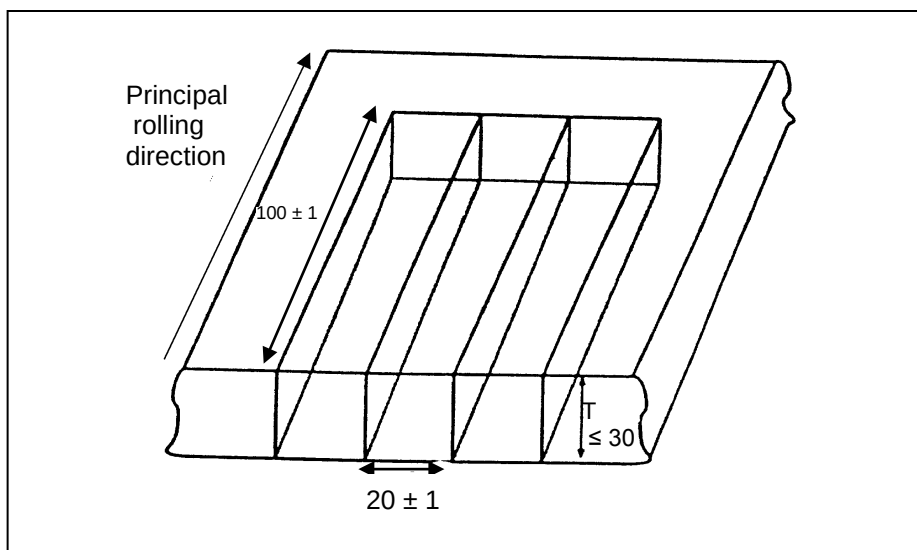


Figure 7: Test specimen location for plates up to 30 mm (1.2 in) thick, inclusive (all dimensions in mm [1 in = 25.4 mm]).

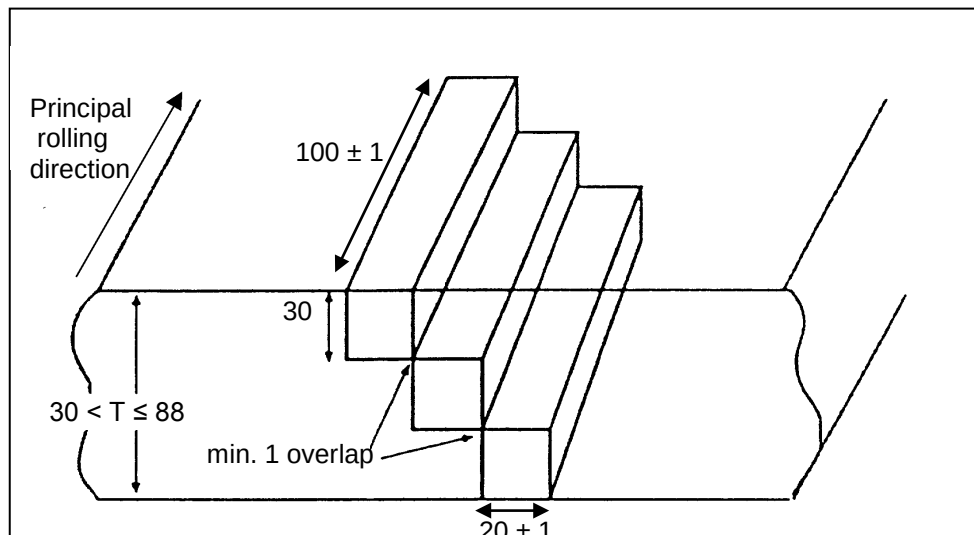


Figure 8: Test specimen location for plates over 30 mm (1.2 in) to 88 mm (3.5 in), inclusive (all dimensions in mm [1 in = 25.4 mm]).

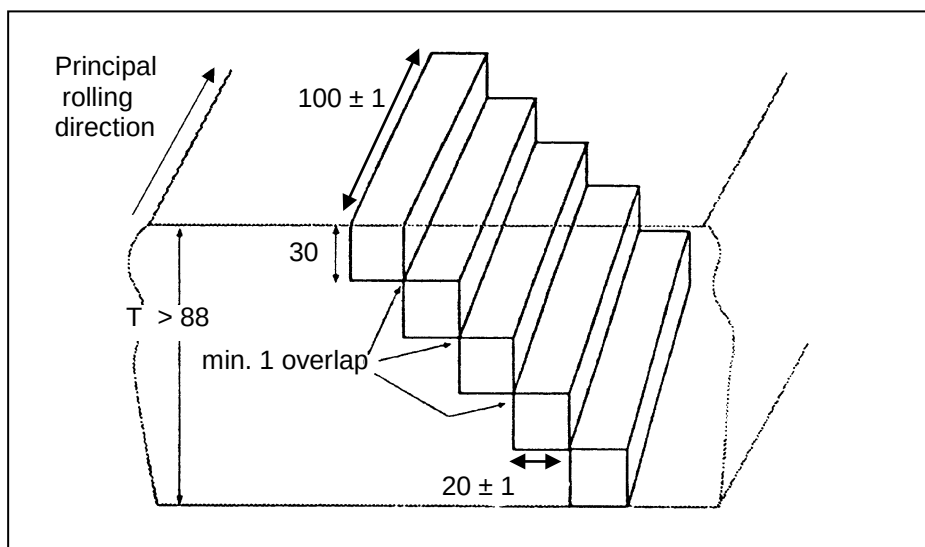


Figure 9: Test specimen location for plates over 88 mm (3.5 in) (all dimensions in mm [1 in = 25.4 mm]).

5.3 Preparation

The preparation of plate test specimens shall be the same as specified for pipe test specimens in Paragraph 4.3.

5.4 Cleaning and Storing

The cleaning and storing of plate test specimens shall be the same as specified for pipe test specimens in Paragraph 4.4.

Section 6: Test Specimens—Fittings

6.1 Size

6.1.1 If fittings are large enough, each test specimen shall be 100 ± 1 mm (4.00 ± 0.04 in) long by 20 ± 1 mm (0.80 ± 0.04 in) wide.

6.1.2 A maximum of 1 mm (0.04 in) shall be removed from each of the original surfaces. Test specimen blanks shall not be flattened.

6.1.3 The test specimen thickness shall be full wall thickness of the fitting up to a maximum of 30 mm (1.2 in). For fittings thicker than 30 mm (1.2 in), the test specimens shall be staggered as described in Section 5.

6.1.4 If a fitting is too small to extract a standard 100 mm (4.00 in) long by 20 mm (0.80 in) wide test specimens, the full size fitting shall be exposed to the test solution.

6.2 Number, Location, and Orientation

6.2.1 The test specimen number, location, and orientation for elbows, tees, reducers, and end caps shall be as described in Table 1.

6.2.2 For end caps, the test specimens shall be extracted longitudinal to the rolling direction of the plate used to manufacture the end cap.

6.2.3 All forged fittings shall have at least one test specimen taken along the forging flash line.

6.2.4 For welded fittings, one test specimen shall be taken across the weld in accordance with Figure 3, and two test specimens shall be taken from the parent metal in a longitudinal orientation at 90 degrees on either side of the weld.

6.2.5 If full size fittings are tested, the number of fittings to be exposed may be either one or three, depending on the fitting size. The number (i.e., one or three) shall be governed by the ability to prepare and examine nine polished sections at the termination of the exposure period.

6.3 Preparation

6.3.1 The preparation of fitting test specimens shall, whenever possible, be the same as specified for pipe test specimens in Paragraph 4.3.

6.3.2 For curved and irregularly shaped test specimens removed from fittings, the two side cut faces of each test specimen shall be ground (wet or dry) and finished with 320 grit paper.

6.3.3 For curved and irregularly shaped test specimens removed from fittings, the two 20 mm (0.80 in) wide faces of each test specimen shall be either ground (wet or dry) to an equivalent 320 grit paper finish or grit blasted to a uniform near-white metal finish in accordance with NACE No. 2/SSPC⁽⁴⁾-SP 10⁹ or ISO 8501-1, Grade Sa 2½.¹⁰

6.3.4 For full size fittings, the complete fitting shall be grit blasted to a uniform near-white metal finish in accordance with NACE No. 2/SSPC-SP 10 or ISO 8501-1, Grade Sa 2½.

6.4 Cleaning and Storage

The cleaning and storage of fitting test specimens shall be the same as specified for pipe test specimens in Paragraph 4.4.

⁽⁴⁾ The Society for Protective Coatings (SSPC), 40 24th St., 6th Floor, Pittsburgh, PA 15222-4656.

Table 1
Number, Location, and Orientation of Test Specimens For Fittings

Fitting Type	No. of Test Specimens	Orientation of Test Specimens	Location of Test Specimens
45 degree elbow	3	Longitudinal	One test specimen shall be machined from the extrados and two test specimens shall be machined from the neutral axis at 180 degree locations. All test specimens shall be machined from mid-length of fitting.
90 degree elbow	3	Longitudinal	One test specimen shall be machined from the extrados and two test specimens shall be machined from the neutral axis at 180 degree locations. All test specimens shall be machined from mid-length of fitting.
Long/short radius return	3	Longitudinal	One test specimen shall be machined from the extrados and two test specimens shall be machined from the neutral axis at 180 degree locations. All test specimens shall be machined from mid-length of fitting.
Concentric reducer	3	Longitudinal	Three test specimens shall be machined from the mid-length location at 120 degree intervals around the circumference.
Eccentric reducer	3	Longitudinal	One test specimen shall be machined from the maximum change in the section (0 degree) and two test specimens shall be machined at 90 degrees on either side at 90 degree and 270 degree locations. All test specimens shall be machined from mid-length of fitting.
Straight tee	3	Longitudinal	Test specimens shall be machined at 90 degree (side), 180 degree (back), and 270 degree (side) locations relative to the outlet. All test specimens shall be machined from mid-length of fitting.
Reducing outlet tee	3	Longitudinal	Test specimens shall be machined at 90 degree (side), 180 degree (back), and 270 degree (side) locations relative to the outlet. All test specimens shall be machined from mid-length of fitting.
Stub end	3	Longitudinal	Test specimens shall be machined from the mid-length location at 120 degree intervals around the circumference.
End cap	3	Radial	One test specimen shall be taken from the center of the top face in a radial direction. One test specimen shall be taken from the top face to side face (knuckle) transition region. One test specimen shall be taken from the side face. (NOTE: For small fittings, the two separate test specimens from the knuckle and side face regions shall incorporate the side face and knuckle region.)

Section 7: Test Specimens—Flanges

7.1 Size

7.1.1 If flanges are large enough, each test specimen shall be 100 ± 1 mm (4.00 ± 0.04 in) long by 20 ± 1 mm (0.80 ± 0.04 in) wide.

7.1.2 A maximum of 1 mm (0.04 in) shall be removed from each of the surfaces. Test specimen blanks shall not be flattened.

7.1.3 The test specimen thickness shall be the full wall thickness of the flange up to a maximum of 30 mm (1.2 in). For flanges thicker than 30 mm (1.2 in), the test specimens shall be staggered as described in Section 5.

7.1.4 If the flange size is too small to extract standard 100 mm (4.00 in) long by 20 mm (0.80 in) wide test specimens, full size flanges shall be exposed to the test solution.

7.2 Number, Location, and Orientation

7.2.1 The test specimen number, location, and orientation for blind flanges and weld neck flanges are described in Table 2.

7.2.2 If full size flanges are tested, the number of flanges to be exposed may be either one or three, depending on the fitting size. The number (i.e., one or three) shall be governed by the ability to prepare and examine nine polished sections at the termination of the exposure period.

7.3 Preparation

7.3.1 The preparation of flange test specimens shall, whenever possible, be the same as specified for pipe test specimens in Paragraph 4.3.

7.3.2 For curved and irregularly shaped test specimens removed from flanges, the two side cut faces of each test specimen shall be ground (wet or dry) and finished with 320 grit paper.

7.3.3 For curved and irregularly shaped test specimens removed from flanges, the two 20 mm (0.80 in) wide faces of each test specimen shall be either ground (wet or dry) to an equivalent 320 grit paper finish or grit blasted to a uniform near-white metal finish in accordance with NACE No. 2/SSPC-SP 10 or ISO 8501-1, Grade Sa 2½.

7.3.4 For full size flanges, the complete flange shall be grit blasted to a uniform near-white metal finish in accordance with NACE No. 2/SSPC-SP 10 or ISO 8501-1, Grade Sa 2½.

7.4 Cleaning and Storage

The cleaning and storage of flange test specimens shall be the same as specified for pipe test specimens in Paragraph 4.4.

Table 2
Number, Location, and Orientation of Test Specimens for Flanges

Flange Type	No. of Test Specimens	Orientation of Test Specimens	Location of Test Specimens
Blind flange	3	Tangential	A blind flange is a solid disc used to close the end of an open flange. It can be forged or manufactured from rolled plate. The method of manufacture and orientation shall be determined prior to sampling. Test specimens shall be machined in the longitudinal orientation from blind flanges manufactured from rolled plate in accordance with Section 5. For blind flanges manufactured from forgings, radial test specimens shall be machined from the 0 degree, 120 degree, and 240 degree locations, following the sampling details in Section 5.
Weld neck flange	3	Longitudinal	Test specimens shall be machined from the weld neck region at 120 degree intervals around the circumference.

Section 8: Test Procedure

8.1 Test Specimen Exposure

8.1.1 Test specimens shall be placed in the test vessel with the wide faces vertical and separated from the test vessel and other test specimens by glass or other nonmetallic rods with a minimum diameter of 6 mm (0.2 in). The longitudinal axis of the test specimens may be either vertical or horizontal. See Figure 10.

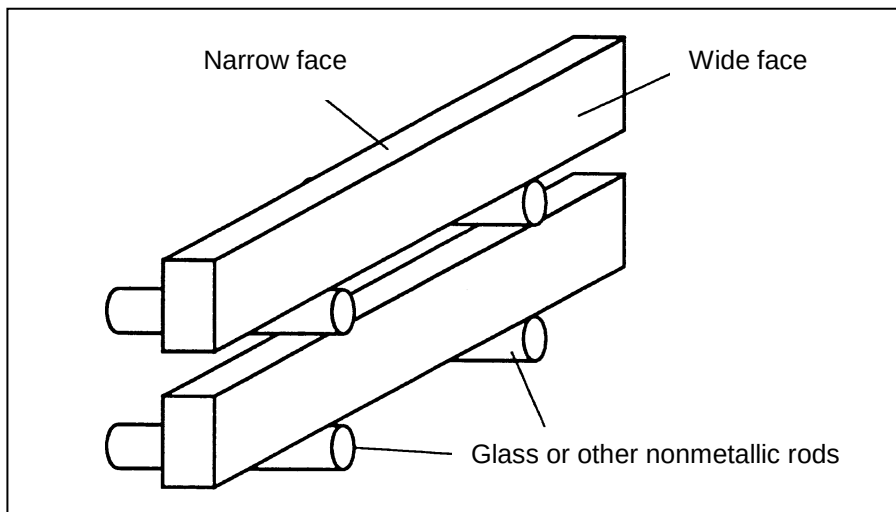


Figure 10: Orientation of test specimens in the test vessel.

8.1.2 The ratio of the volume of test solution to the total surface area of the test specimens shall be a minimum of 3 mL/cm². As long as the specified ratio of volume of test solution to test specimen surface area is maintained, as many test specimens as will fit in the test vessel fully submerged and without touching may be exposed at one time.

8.1.3 If Test Solution A is used, the test solution shall be prepared in a separate sealed vessel that is purged with nitrogen for at least one hour at a rate of 100 cm³/min per liter of test solution prior to transferring the test solution to the test vessel, which has been subject to nitrogen purging in advance. The test solution shall consist of 5.0 wt% NaCl and 0.50 wt% CH₃COOH in distilled or deionized water (i.e., 50.0 g of NaCl and 5.00 g of CH₃COOH dissolved in 945 g of distilled or deionized water). The initial pH shall be 2.7 ± 0.1. All reagents added to the test solution shall be measured to ± 1.0% of the quantities specified.

8.1.4 If Test Solution B is used, the test vessel shall be prepared in a separate sealed vessel that is purged with nitrogen for at least one hour at a rate of 100 cm³/min per liter of test solution prior to transferring the test solution to the test vessel, which has been subject to nitrogen purging in advance. The test solution shall consist of synthetic seawater prepared in accordance with Paragraph 2.4. The initial pH shall be in the range of 8.1 to 8.3 for the test to be valid.

8.2 Purging and Introduction of H₂S

8.2.1 The nitrogen purge gas and H₂S gas shall be introduced near the bottom of the test vessel.

8.2.2 The sealed test vessel shall be purged of air with nitrogen for at least one hour at a rate of 100 cm³/min per liter of test vessel volume. Purging shall begin immediately after the test vessel is filled and shall be done at a rate of at least 100 cm³/min per liter of test solution.

8.2.3 After purging, H₂S gas shall be bubbled through the test solution. The rate of bubbling shall be at least 200 cm³/min per liter of test solution for the first 60 minutes; thereafter, a positive pressure of H₂S gas must be maintained at a constant gas flow rate to ensure that the test solution remains saturated with H₂S. The concentration of H₂S in the test solution shall be measured by iodometric titration and shall be a minimum of 2,300 ppm at the end of the test. An acceptable iodometric titration procedure is detailed in Appendix C (nonmandatory).

8.3 pH Measurement

8.3.1 pH at start of test—If Test Solution A is used, the pH at the start of the test shall be measured immediately after H₂S saturation and shall be within the range of 2.7 to 3.3. If Test Solution B is used, the pH shall be measured immediately after H₂S saturation and shall be within the range of 4.8 to 5.4.

8.3.2 pH at end of test—At the end of the test, the pH of the test solution shall be measured. For Test Solution A, the pH shall not exceed 4.0 for the test to be valid. For Test Solution B, the pH shall be within the range of 4.8 to 5.4 for the test to be valid.

8.4 Test Duration

The test duration shall be 96 hours. The test time shall begin immediately upon completion of the initial 60 minute H₂S introduction period (see Paragraph 8.2.3).

8.5 Test Temperature

The temperature of the test solution during contact with the test specimens shall be 25 ± 3 °C (77 ± 5 °F).

Section 9: Evaluation of Test Specimens

9.1 After testing, each exposed test specimen shall be cleaned to remove scale and deposits. Exposed test specimens may be cleaned with detergent and a wire brush or may be lightly sandblasted. Exposed test specimens must not be cleaned with acid or by any other means that might promote hydrogen absorption.

9.2 After each exposed test specimen has been cleaned, it shall be sectioned for examination as follows:

9.2.1 Each pipe test specimen shall be sectioned for examination as shown in Figures 2 through 6.

9.2.2 Each plate test specimen shall be sectioned for examination as shown in Figure 2.

9.2.3 Each fitting test specimen shall be sectioned for examination as shown in Figure 2. For welded fittings, each weld area test specimen shall be sectioned for examination as shown in Figure 3.

9.2.4 Each full size fitting test specimen shall be sectioned for examination at three equally spaced locations, with the sectioned faces being oriented transverse to the longitudinal axis. For end cap fittings, the section shall be cut 20 mm (0.80 in) in width and shall cover the side face, top face, and top face to side face (knuckle) transition regions normal to the rolling direction.

9.2.5 Each flange test specimen shall be sectioned for examination as shown in Figure 2. For welded flanges, each weld area test specimen shall be sectioned for examination as shown in Figure 3.

9.2.6 Each full size flange test specimen shall be sectioned for examination at three equally spaced locations along the neck region, with the sectioned faces being oriented transverse to the longitudinal axis.

9.3 Each section shall be polished metallographically and etched, if necessary, so that cracks can be distinguished from small inclusions, laminations, scratches, or other discontinuities. Only a light etch shall be used; a heavy etch may obscure small cracks. A metallographic preparation method that does not smear the metal surfaces such that significant cracks may become invisible shall be used. Therefore, all faces to be examined shall be subjected to either wet magnetic particle testing or macroetching prior to final metallographic polishing. Alternatively, a preparation procedure that is described in detail and has proved to result in clearly visible cracks (if present) after final polishing may be used.

9.4 Cracks shall be measured as illustrated in Figure 11. In measuring crack length and thickness, cracks separated by less than 0.5 mm (0.002 in) shall be considered a single crack. When cracks in curved sections or ring sections are measured, particularly in full size fitting test specimens, the curvature of the section shall be taken into account. All identifiable cracks visible at magnifications up to 100X shall be included in the calculation, except those that lie entirely within 1.0 mm (0.04 in) of the internal or external surface of the test specimen. (It may be necessary to examine some sections at higher magnifications to distinguish between small cracks, inclusions, pits on the side surfaces, or other discontinuities.)

9.5 The crack sensitivity ratio (CSR), crack length ratio (CLR), and crack thickness ratio (CTR) shall be calculated in accordance with Equations (1), (2), and (3) respectively and reported for each section and the average for each test specimen.

$$CSR = \frac{\sum (a \times b)}{(W \times T)} \times 100\% \quad (1)$$

$$CLR = \frac{\sum a}{W} \times 100\% \quad (2)$$

$$CTR = \frac{\sum b}{T} \times 100\% \quad (3)$$

Where:

- a = crack length
- b = crack thickness
- W = section width
- T = test specimen thickness

NOTE: In the past, CSR has been calculated by some investigators as $(\sum a \times \sum b)/(W \times T)$, which is simply the product of CLR x CTR (i.e., $\sum a/W \times \sum b/T$); it does not give the same value as $\sum (a \times b)/(W \times T)$.

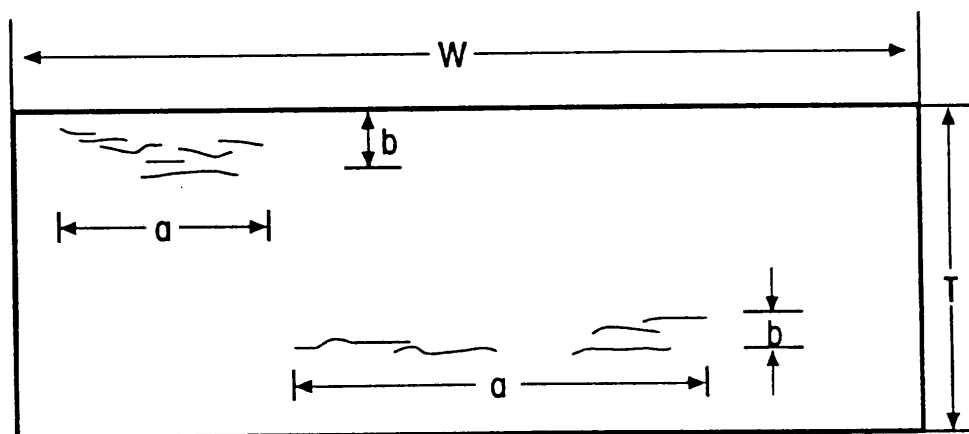


Figure 11: Test specimen and crack dimensions to be used in calculating CSR, CLR, and CTR.

Section 10: Reporting Test Results

10.1 The type, grade, and manufacturing method of the pipe, plate, fitting, or flange shall be reported (e.g., API⁽⁴⁾ 5L,¹¹ Grade X52, seamless; ASTM A53,¹² Grade B, ERW; ASTM A516,¹³ Grade 70; ASTM A234,¹⁴ Grade WPB; ASTM A105,¹⁵ Grade B; ASTM A350,¹⁶ Grade LF2, etc.). Manufacturer, chemical composition, heat treatment, mechanical properties, and processing data shall be included, if available.

10.2 The following shall be reported:

- (a) Method of testing for adequacy of test specimen degreasing;
- (b) Test solution used (Test Solution A or Test Solution B [Stock Solutions No. 1 and No. 2]);
- (c) pH of the test solution before introduction of H₂S—specified in Paragraphs 8.1.3 and 8.1.4;
- (d) pH of the test solution at start of test (after saturation with H₂S)—specified in Paragraph 8.3.1;
- (e) pH of the test solution at end of test—specified in Paragraph 8.3.2; and
- (f) Temperature of the test solution—specified in Paragraph 8.5.

10.3 Any test condition and procedure not in accordance with this standard shall be reported.

⁽⁴⁾ American Petroleum Institute (API), 1220 L St. NW, Washington, DC 20005-4070.

10.4 The individual CSR, CLR, and CTR shall be reported for each of the three sections from each test specimen, and the average CSR, CLR, and CTR shall be reported for each test specimen and for all test specimens from the sample. NOTE: A sample is defined as a set of test specimens.

10.5 For small-diameter, thin-wall ERW and seamless pipe, the actual wall thickness as well as the test specimen thickness as a percentage of the pipe wall thickness shall be reported.

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17. OSHA⁽⁵⁾ Rules and Regulations, 29 CFR 1910.1000 (Washington, DC: OSHA, 1996).
18. Chemical Safety Data Sheet SD-36 (Washington, DC: Manufacturing Chemists Association,⁽⁶⁾ 1950).
19. N. Irving Sax, *Dangerous Properties of Industrial Materials* (New York, NY: Reinhold Book Corp., 1984).

Appendix A Safety Considerations in Handling H₂S Toxicity (Nonmandatory)

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

H₂S is perhaps responsible for more industrial poisoning accidents than is any other single chemical. A number of these accidents have been fatal. H₂S must be handled with caution and any experiments using it must be planned carefully. The OSHA maximum allowable concentration of H₂S in the air for an eight-hour work day is 20 parts per million (ppm), well above the level detectable by smell.¹⁷ However, the olfactory nerves can become deadened to the odor after exposure of 2 to 15 minutes, depending on concentration, so that odor is not a reliable alarm system.

Briefly, the following are some of the human physiological reactions to various concentrations of H₂S. Exposure to concentrations in the range of 150 to 200 ppm for prolonged periods may cause edema of the lungs. Nausea, stomach distress, belching, coughing, headache, dizziness, and blistering are symptoms of poisoning in this range of concentration. Pulmonary complications, such as pneumonia, are strong possibilities from such subacute exposure. At 500 ppm, unconsciousness may occur in less than 15 minutes, and death within 30 minutes. At concentrations above 1,000 ppm, a single inhalation may result in instantaneous unconsciousness, complete respiratory failure, cardiac arrest, and death.

Additional information on the toxicity of H₂S can be obtained from the Chemical Safety Data Sheet SD-36¹⁸ and from *Dangerous Properties of Industrial Materials*.¹⁹

Fire and Explosion Hazards

H₂S is a flammable gas and yields poisonous sulfur dioxide (SO₂) as a combustion product. In addition, its explosive limits range from 4 to 46% in air. Appropriate precautions shall be taken to prevent these hazards from developing.

Safety Procedures During Test

All tests shall be performed in a hood with adequate ventilation to exhaust all of the H₂S. The H₂S flow rates during the test should be kept low to minimize the quantity exhausted. A 10% caustic absorbent solution for effluent gas can be used to further minimize the quantity of H₂S gas exhausted. This caustic solution needs periodic replenishing. Provision shall be made to prevent backflow of the caustic solution into the test vessel if the H₂S flow is interrupted. Suitable safety equipment shall be used when working with H₂S.

Because the downstream working pressure frequently rises as corrosion products, debris, etc., accumulate and interfere with regulation at low flow rates, particular attention should be given to the output pressure on the pressure regulators. Gas cylinders shall be securely fastened to prevent tipping and breaking of the cylinder head. Because H₂S is in liquid form in the cylinders, the high-pressure gauge must be checked frequently, because relatively little time elapses after the last liquid evaporates and the pressure drops from 1.7 MPa (250 psig) to atmospheric pressure. The cylinder 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 H₂S and move upstream into the flowline, regulator, and even the cylinder. A check valve in the line should prevent the problem if the valve works properly. However, if such an accident occurs, the remaining H₂S should be vented as rapidly and safely as possible and the manufacturer notified so that the cylinder can be given special attention.

⁽⁵⁾ Occupational Safety and Health Administration (OSHA), 200 Constitution Ave. NW, Washington, DC 20210.

⁽⁶⁾ American Chemistry Council (ACC) (formerly known as the Manufacturing Chemists Association and then as the Chemical Manufacturers Association), 700 Second St. NE, Washington, DC 20002.

Appendix B Explanatory Notes on Test Method (Nonmandatory)

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

Reasons for Reagent Purity

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

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

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

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 environmental cracking) 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 environmental cracking, 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.

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 HIC test results.

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.

Appendix C
Determination of H₂S Concentration in Test Solution by Iodometric Titration
(Nonmandatory)

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

This procedure details an acceptable method that may be used for the determination of the of H₂S concentration in the test solution by iodometric titration.

Test Reagents

Standard iodine solution (0.1 N⁽⁷⁾)
Concentrated HCl (approx. 37 wt% HCl)
Starch solution
Standard sodium thiosulfate solution (0.1 N⁽⁷⁾)

"Analar" grade chemicals shall be used.

Test Equipment

10 mL and 25 mL pipettes
250 mL conical flask with stopper
100 mL beaker
25 mL burette

Procedure

1. Pipette 25 mL⁽⁷⁾ of standard iodine solution (0.1 N⁽⁷⁾) into a 250 mL conical flask.
2. Acidify with approximately 5 mL of concentrated HCl.
3. Transfer approximately 50 mL of test solution from the test vessel into a clean beaker, discarding the initial 25 to 50 mL.
4. Immediately extract 10 mL of test solution from the beaker via a pipette and discard. Pipette a further 10 mL⁽⁷⁾ and transfer this sample to the conical flask.
5. Titrate with the standard sodium thiosulfate solution (0.1 N⁽⁷⁾) until the solution changes color from dark yellow/tan to pale yellow.
6. Add approximately 2 mL of starch solution.
7. Continue to titrate with the standard sodium thiosulfate solution (0.1 N⁽⁷⁾) until the end point changes from dark blue to pale straw.
8. Calculate the H₂S concentration (ppm) using Equation (C1):

⁽⁷⁾ The following standard solution concentrations and volumes should be used for H₂S-saturated solutions containing 2,300 ppm to 3,500 ppm H₂S:

0.1 N iodine solution.
0.1 N sodium thiosulphate solution
25 mL iodine solution volume
10 mL test sample volume

If the procedure is used to measure the H₂S concentration in test solutions saturated with less than 2,300 ppm H₂S, the above values may need to be adjusted to maintain accuracy.

$$\text{H}_2\text{S concentration (ppm)} = \frac{[(A \times \text{Factor}) - B]}{C} \times 17,030 \quad (\text{C1})$$

Where: A = Normality of standard iodine solution times the volume used (mL)
 B = Normality of standard sodium thiosulfate solution times the volume used (mL)
 C = Volume of sample (mL)
 Factor = Standardization factor for standard iodine solution (see *Standardization* below)

Standardization

The iodine and sodium thiosulfate solutions must be standardized when they are prepared for use in this determination.

1. To standardize these solutions, pipette 25 mL of the 0.1 N iodine solution into a conical flask and titrate with the 0.1 N sodium thiosulfate solution to a clear end point.
2. Record the result.
3. Calculate the "Factor" for the H₂S concentration calculation using Equation (C2):

$$\text{Factor} = \frac{B}{A} \quad (\text{C2})$$

Where: A = Normality of iodine solution times the volume used (mL)
 B = Normality of sodium thiosulfate solution times the volume used (mL)

4. If the factor is within the limits of 0.95 to 1.05, use the value of the Factor calculated in Equation (C2) in Equation (C1). These solutions are acceptable.

If the factor is outside the set limits of 0.95 to 1.05, replace the iodine solution and repeat the standardization.

Measurement Uncertainty

Refer to Equation (C3) for the measurement uncertainty for the determination, based on a H₂S concentration of 2,500 ppm:

$$95\% \text{ confidence interval} = \pm 4.2\% \quad (\text{C3})$$

Example: If a 10 mL sample mixed with 25 mL of 0.1 N iodine solution was titrated to the end point with 10 mL of 0.1 N sodium thiosulfate solution, with a factor of 1.00, the 95% confidence interval (CI) can be calculated as shown in Equation (C4). Note that the result is $\pm 4.2\%$ of the H₂S concentration calculated in accordance with Equation (C1).

$$95\% \text{ CI} = \frac{[(2.5 \times 1.0) - 1.00]}{10} \times 17,030 \times 4.2\% \quad (\text{C4})$$

$$95\% \text{ CI} = \pm 107 \text{ ppm}$$

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