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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, “Stepwise Cracking of Pipeline Steels,” 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 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. In the 2016 revision of this standard, Fitness-For-Purpose testing in an alternative test solution, to be used with gas containing mixtures of H_2S and CO_2, was included to assess HIC damage under mildly sour test conditions with reduced partial pressure of H_2S in a range of pH values.

Test conditions are not designed to simulate any particular pipeline or process operation, even though in Fitness-for-Purpose tests, partial pressures of H_2S and pH values appropriate to the intended application must be selected. The test is intended to evaluate resistance to HIC only, and not to 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 TG T-1F-20 in 1984. It was revised in 1996 by TG T-1F-20, and in 2003, 2011, and 2016 by TG 082, “Stepwise Cracking of Pipeline Steels,” which is administered by Specific Technology Group (STG) 32, “Oil and Gas Production—Metallurgy” and sponsored by STG 34, “Petroleum Refining and Gas Processing,” and STG 62, “Corrosion Monitoring and Measurement—Science and Engineering Applications.” 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
Test Method
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Contents

Figure

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 three standard test solutions—Test Solution A, an acidified brine solution consisting of sodium chloride (NaCl) and acetic acid (CH_3COOH) dissolved in distilled or deionized water saturated with H_2S at ambient temperature and pressure; or Test Solution B, a synthetic seawater solution saturated with H_2S at ambient temperature and pressure; or Test Solution C, a buffered solution consisting of sodium chloride (NaCl) and sodium acetate (CH_3COONa) dissolved in distilled or deionized water saturated with gas containing mixtures of H_2S and CO_2 at ambient temperature and pressure enabling testing to be conducted at different H_2S partial pressures in the range 0.001 to 1 bar. After a specified time, the test specimens are removed and evaluated.

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.

1.3 In Fitness-for-Purpose testing, the test environment and partial pressures of gases appropriate to the intended application are selected.

NOTE: The test conditions do not duplicate all aspects of service conditions, for example temperature, but will allow sufficient discrimination of the applicability of candidate steels. See Paragraph 8.1.5 and associated notes.

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]

1.5 For additional information, the presence or absence of HIC in the exposed specimens may be evaluated by automated ultrasonic testing prior to metallographic sectioning and examination. A procedure is provided in Appendix A (nonmandatory).

Section 2: Reagents

2.1 The reagents for Test Solution A shall be an inert gas (nitrogen, argon, or other suitable non-reactive gas) for purging, H_2S gas, NaCl, CH_3COOH, and distilled or deionized water. The reagents for Test Solution B shall be an inert gas for purging, H_2S gas, and synthetic seawater. The reagents for Test Solution C shall be an inert gas for purging, a mixture of H_2S and carbon dioxide (CO_2), with H_2S content sufficient to produce the specified H_2S partial pressure, NaCl, CH_3COONa, hydrochloric acid (HCl) or sodium hydroxide (NaOH) added to achieve the specified pH and distilled or deionized water.

NOTE: H_2S is highly toxic and must be handled with caution. See Appendix B (nonmandatory).

2.2 The NaCl, CH_3COOH, CH_3COONa, HCl and NaOH shall be reagent grade chemicals.

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

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

2.3 The inert gas purity shall be 99.998% or greater. The H_2S gas shall be chemically pure (CP grade) 99.5% minimum purity. Test gas mixtures consisting of H_2S and CO_2 should be contained in a standard gas cylinder 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. The test water shall be distilled or deionized and of quality equal to or greater than ASTM D1193 Type IV. See Appendix C (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).

Section 3: Testing Apparatus

3.1 Figure 1 is a schematic diagram of a typical test assembly (not to scale).

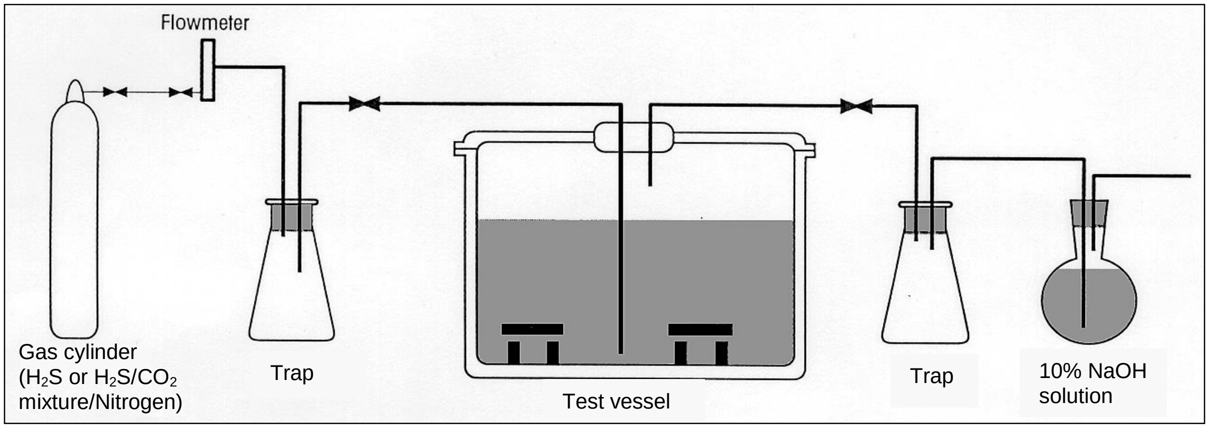

Figure 1: Schematic Diagram of Typical Test Assembly

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 H_2S or H_2S containing gas mixtures.

NOTE: In Fitness-for-Purpose HIC testing, a homogeneous test solution is required to facilitate pH control. In large test vessels, this may be achieved by continuous stirring of the test solution throughout the test. For Fitness-for-Purpose tests where buffering is less strong, stirring of the solution may also limit an increase in pH local to the corroding steel surface.

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.

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

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.

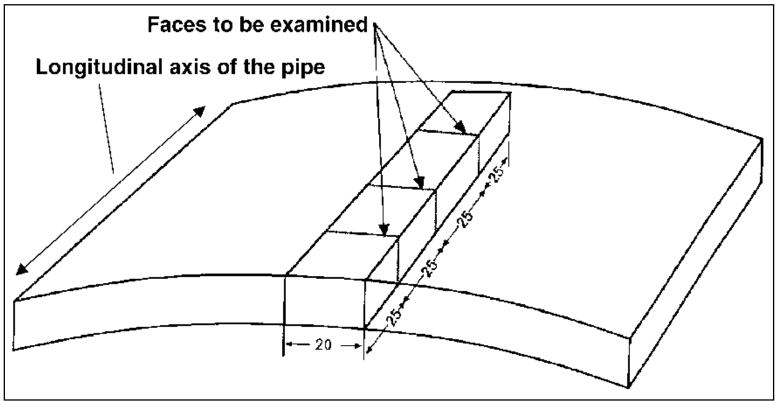


Figure 2: Seamless Pipe and Parent Metal of Longitudinally Welded Pipe (All Dimensions in mm [1 in = 25.4 mm])

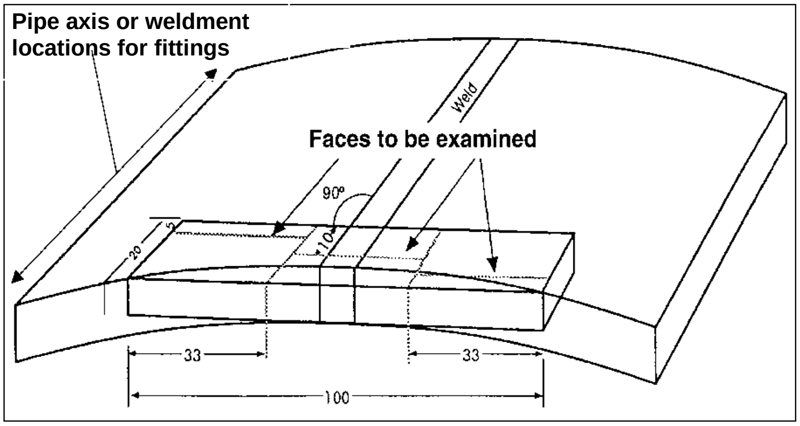
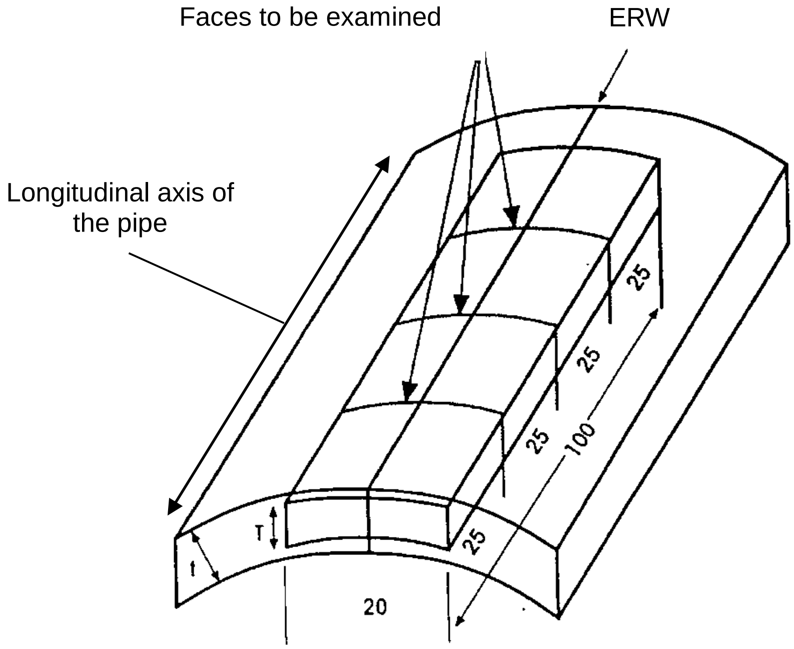
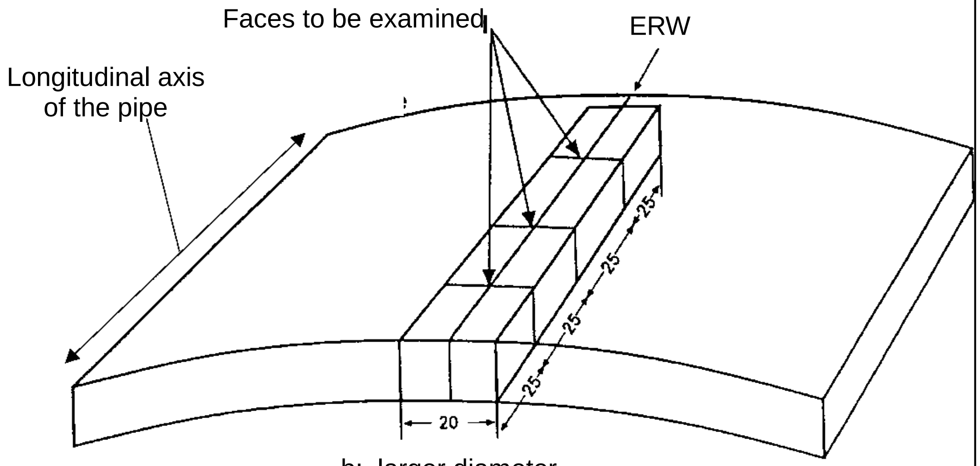


Figure 3: Weld Area of Longitudinally Welded Pipe or Welded Fittings (All Dimensions in mm [1 in = 25.4 mm])



a: small diameter, thin wall



b: larger diameter

Figure 4: Weld Area of ERW Pipe (All Dimensions in mm [1 in = 25.4 mm])

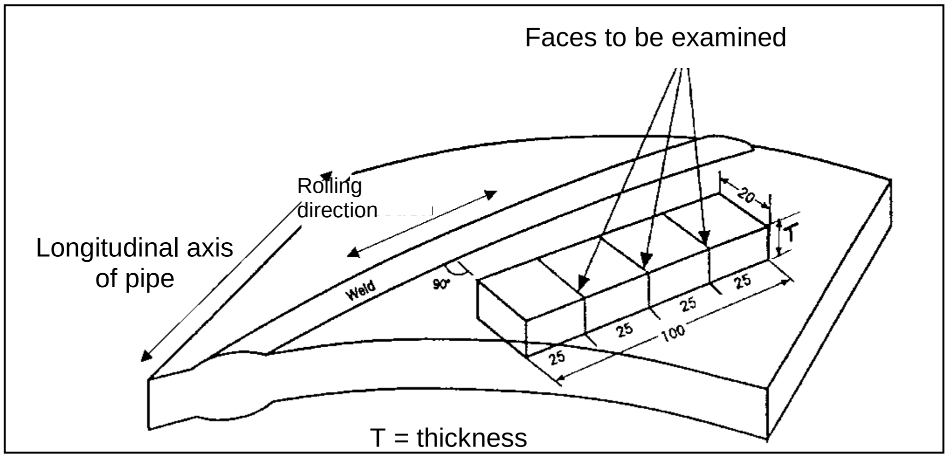


Figure 5 (a): **Parent Metal of Spiral-Welded Pipe (All Dimensions in mm [1 in = 25.4 mm])**

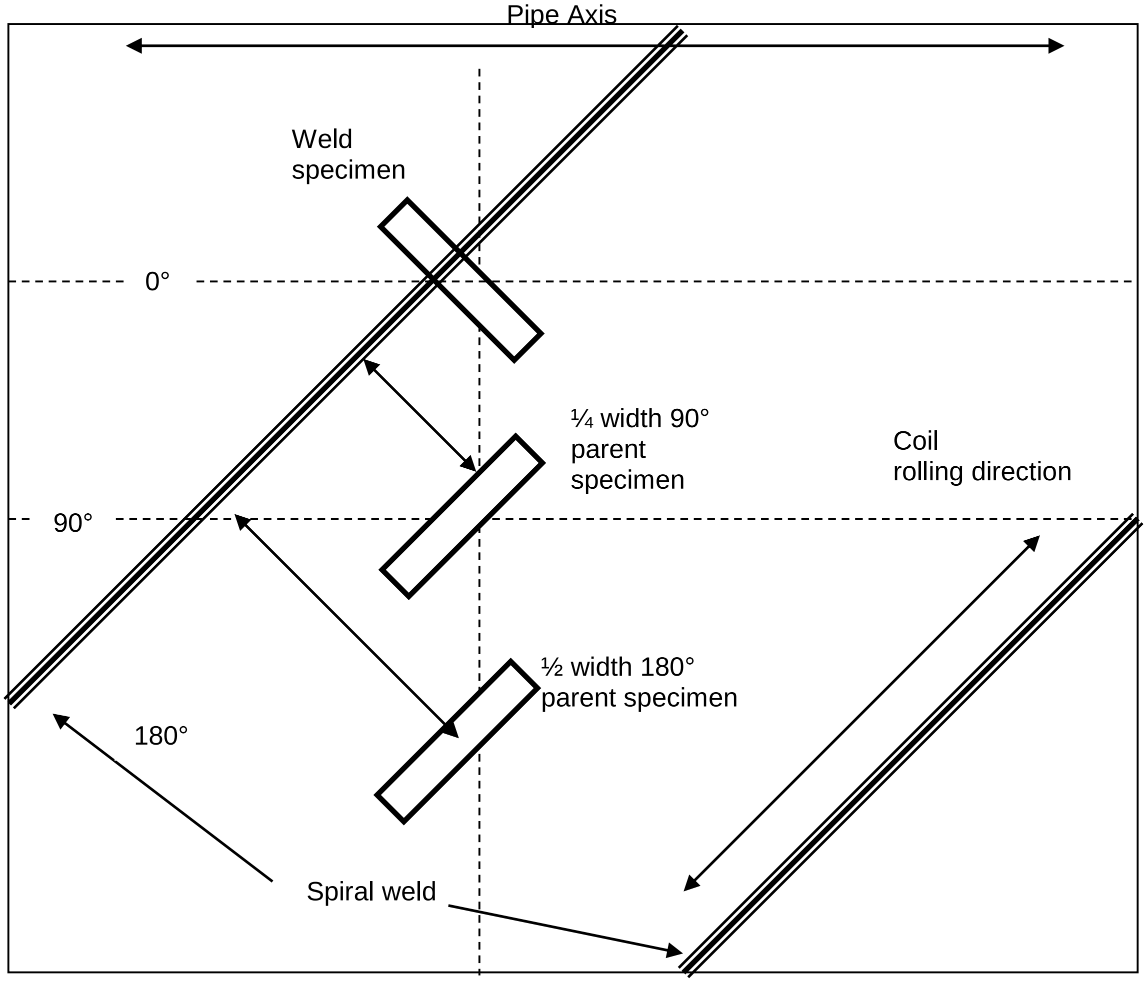


Figure 5 (b): Location and Orientation of Test Specimens to Be Taken from Spiral-Welded Pipe

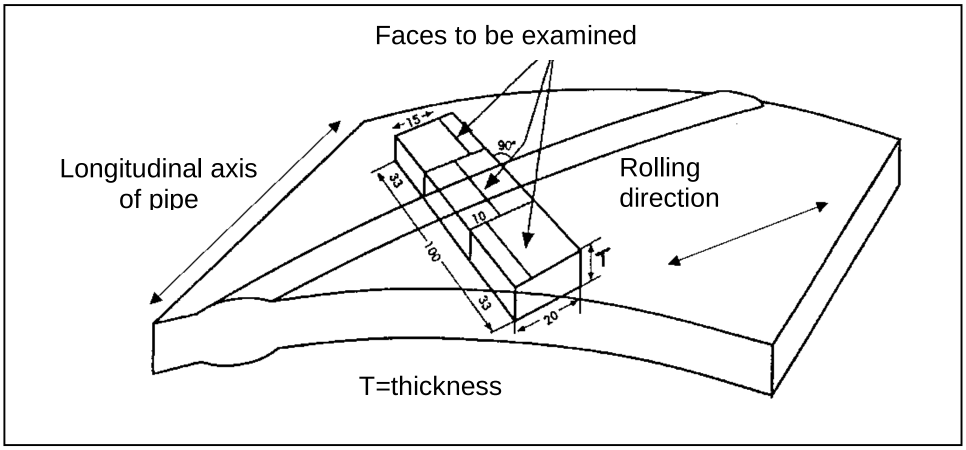


Figure 6: Weld Area of Spiral-Welded Pipe (All Dimensions in mm [1 in = 25.4 mm])

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 either machined and/or ground (wet or dry) to an equivalent 320 grit paper finish. For machining, the last two passes shall be such that a maximum of 0.05 mm (0.002 in) of material is removed.

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. Each test specimen shall be either machined and/or 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 10[9] or ISO 8501-1, Grade Sa 2½.[10] 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 F21[8] 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

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(4) The Society for Protective Coatings (SSPC), 800 Trumbull Drive, Pittsburgh, PA 15205.

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 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.

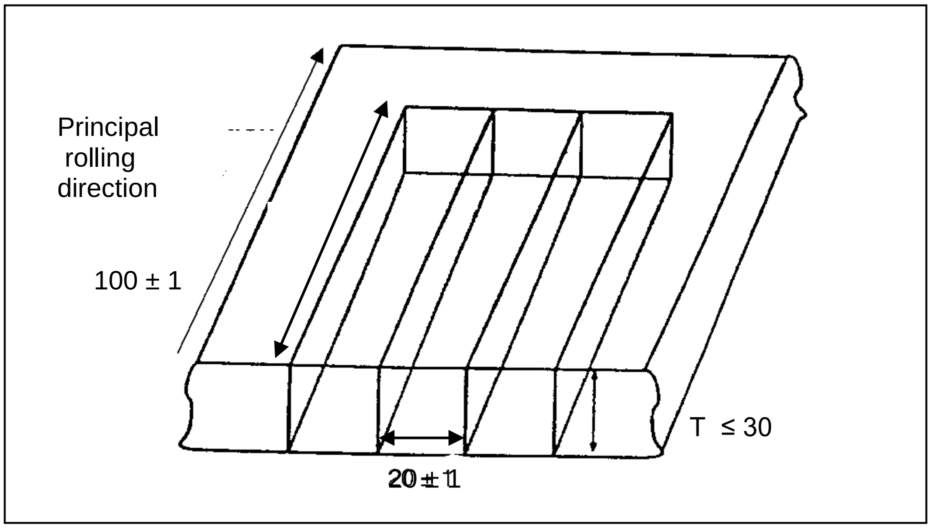

Figure 7: Test Specimen Location for Plates up to 30 mm (1.2 in) Thick, Inclusive (All Dimensions in mm [1 in = 25.4 mm])

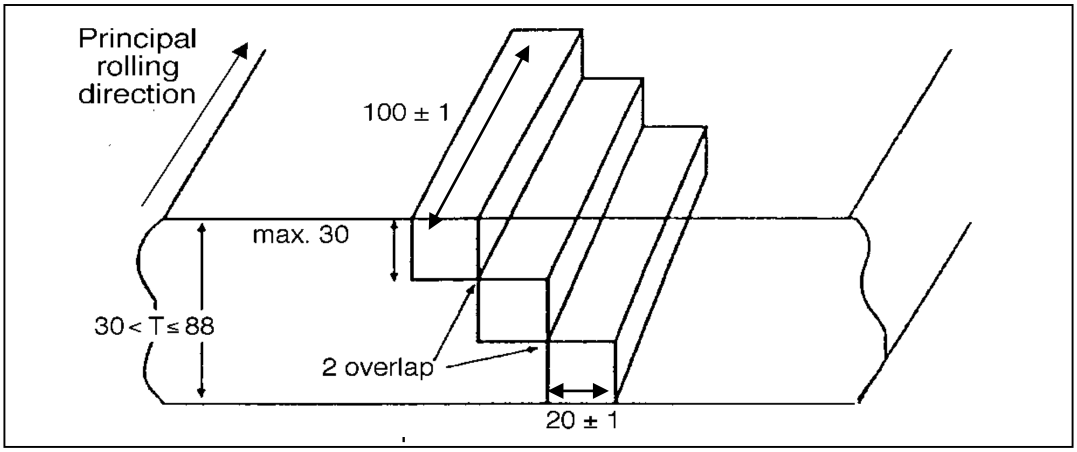

Figure 8: Test Specimen Location for Plates 30 mm (1.2 in) to 88 mm (3.5 in) Thick, Inclusive (All Dimensions in mm [1 in = 25.4 mm])

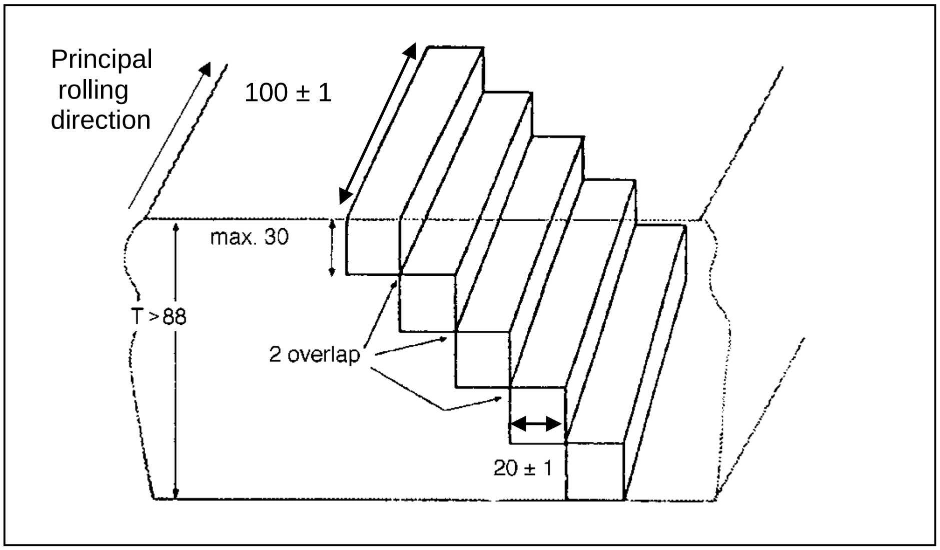

Figure 9: Test Specimen Location for Plates over 88 mm (3.5 in) Thick (All Dimensions in mm [1 in = 25.4 mm])

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 specimen, 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-SP 10 or ISO 8501-1, Grade Sa 2½.

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.

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 specimen~~s~~ 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.

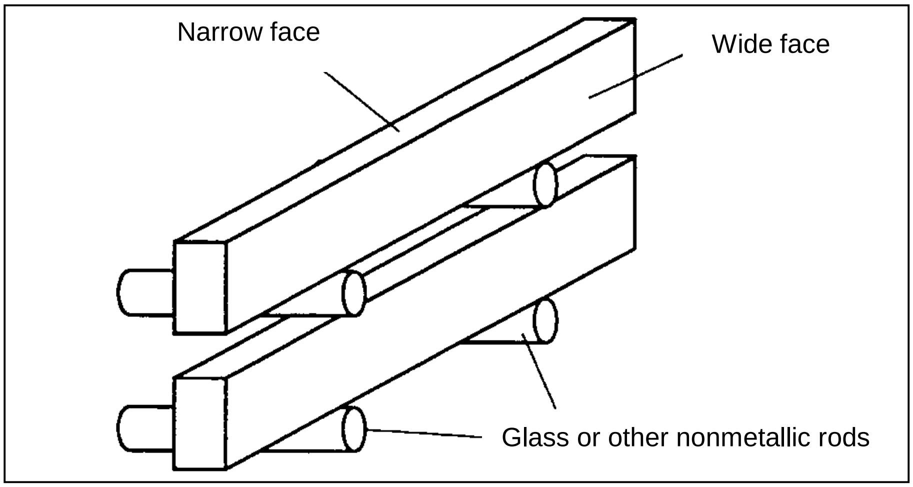

Figure 10: Orientation of Test Specimens in the Test Vessel

8.1.2 If Test Solution A or B is used, the ratio of the volume of test solution to the total surface area of the test specimens shall be a minimum of 3 mL/cm^2. If Test Solution C is used, the ratio of the volume of test solution to the total surface area of the test specimens shall be a minimum of 5 mL/cm^2. 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.

NOTE: For Test Solution C, a ratio of the volume of test solution to the total surface area of the test specimens higher than 5 mL/cm^2 should be used for tests at pH levels below 4.0 to reduce the frequency of re-adjustment of pH to the target pH (see Paragraph 8.3.1). The use of an alternate test solution with greater pH stability may also be appropriate (see Paragraph 8.1.5).

8.1.3 If Test Solution A is used, the test solution shall be prepared in a separate sealed vessel that is purged with inert gas for at least one hour at a rate of 100 mL/min per liter of test solution prior to transferring the test solution to the test vessel, which has been subjected to inert gas purging in advance (see Paragraph 8.2.2). The test solution shall consist of 5.0 wt% NaCl and 0.50 wt% CH_3COOH in distilled or deionized water (i.e., 50.0 g of NaCl and 5.00 g of CH_3COOH 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 solution shall be prepared in a separate sealed vessel that is purged with inert gas for at least one hour at a rate of 100 cm^3/min per liter of test solution prior to transferring the test solution to the test vessel, which has been subjected to inert gas purging in advance (see Paragraph 8.2.2). 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.

8.1.5 If Test Solution C is used, the oxygen concentration in the test solution shall be maintained below 50 ppb. The laboratory shall have a demonstrated and documented procedure for solution deaeration validating that the methodology adopted achieves the required concentration of oxygen. The test solution shall consist of 5.0 wt% NaCl and 0.40 wt% CH_3COONa in distilled or deionized water (i.e. 50.0 g of NaCl and 4.00 g of CH_3COONa dissolved in 946 g of distilled or deionized water). The initial pH shall be adjusted to the target pH ± 0.2 pH units by addition of HCl or NaOH before saturation with the H_2S/CO_2 gas mixture for the test to be valid. All reagents added to the test solution shall be measured to ± 1.0% of the quantities specified.

NOTE: The oxygen concentration in the test vessel may be monitored directly or in a separate test carried out using the same apparatus and procedure, but with an oxygen concentration monitor, to demonstrate that the methodology adopted achieves the required concentration of oxygen.

NOTE: For tests requiring greater pH stability, NACE TM0177 Solution B (0.47 N total acetate) adjusted to the selected test pH value by addition of HCl or NaOH may be more appropriate. Where this solution is selected, it shall be reported as "NACE TM0177 Solution B" quoting the adjusted test pH. An alternate solution with strong buffering capacity proposed by the Iron and Steel Institute of Japan (ISIJ)(5) high-strength line pipe (HLP) research committee,[11,12,13] including high CH_3COOH / CH_3COONa (0.93 N total acetate), may also be appropriate. Where this solution is selected, it shall be reported as "HLP solution pH x.x".

8.2 Purging and Introduction of H_2S or H_2S/CO_2 Gas Mixtures

8.2.1 The inert purge gas and test gas (H_2S or H_2S/CO_2 gas mixture) shall be introduced near the bottom of the test vessel.

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

8.2.3 If Test Solution A or B is used; after purging, H_2S gas shall be bubbled through the test solution. The rate of bubbling should be 200 mL/min per liter of test solution for at least one hour; thereafter, a constant flow of H_2S gas shall be maintained at a sufficient flow rate to ensure that the test solution remains saturated with H_2S for the duration of the test. The concentration of H_2S in the test solution shall be measured by iodometric titration at the start (after saturation) and at the end of the test, and shall be a minimum of 2,300 mg/L. An acceptable iodometric titration procedure is detailed in Appendix D (nonmandatory).

8.2.4 If Test Solution C is used; after purging, the H_2S/CO_2 gas mixture shall be bubbled through the test solution. The rate of bubbling should be 200 mL/min per liter of test solution for at least one hour; thereafter, a constant flow of test gas shall be maintained at a sufficient flow rate to ensure that the test solution remains saturated with the test gas for the duration of the test. The concentration of H_2S in the test solution shall be measured by iodometric titration at the start (after saturation) and at the end of the test, and shall be of the minimum value as calculated from Equation (1), dependent on the mole fraction of H_2S in the test gas. An acceptable iodometric titration procedure is detailed in Appendix D (nonmandatory).

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(5) The Iron and Steel Institute of Japan (ISIJ), Tekko Kaikan (5F), 3-2-10, Nihonbashi-Kayabacho, Chuo-ku, Tokyo 103-0025 Japan.

$$cH_2S = 2300 \times \frac{xH_2S}{100} \qquad (1)$$

where: cH_2S is the concentration of H_2S in the test solution, expressed in mg/L
xH_2S is the mole fraction of H_2S in the test gas, expressed as a percentage

8.3 pH Measurement and Adjustment

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 H_2S 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 H_2S saturation and shall be within the range of 4.8 to 5.4. If Test Solution C is used, the pH at the start of the test shall be measured immediately after saturation with the H_2S/CO_2 mixture and shall be the target pH ± 0.2 pH units. If necessary, the pH shall be re-adjusted to the target pH ± 0.2 pH units by addition of HCl or NaOH. During the test, the pH may alter, but shall not be allowed to change by more than ± 0.2 pH units. This shall be achieved by periodically regenerating the buffering power of the test solution by pH adjustment by addition of HCl or NaOH. In addition, the exclusion of oxygen from the test during pH adjustment shall be ensured. Details of pH adjustment shall be recorded.

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. For Test Solution C, the pH shall be the target pH ± 0.2 pH units for the test to be valid.

8.4 Test Duration

If Test Solution A or B is used, the test duration shall be 96 hours.
If Test Solution C is used, the test duration shall be in accordance with Table 3, dependent on the partial pressure of H_2S in the test gas.

Table 3
Test Duration for HIC Tests in Solution C Dependent on the Partial Pressure of H_2S
(pH_2S: Partial Pressure of H_2S; xH_2S: Mole Fraction of H_2S in the Test Gas)

pH_2S bar	**xH_2S** mole-%	**Test Duration** hours/days
$0.001 \le pH_2S \le 0.003$	$0.1 \le xH_2S \le 0.3$	2160/90
$0.003 < pH_2S \le 0.01$	$0.3 < xH_2S \le 1$	720/30
$0.01 < pH_2S \le 0.1$	$1 < xH_2S \le 10$	336/14
$0.1 < pH_2S$	$10 < xH_2S$	96/4

NOTE: The test parameters given in Table 3 for HIC tests in Solution C have been chosen in accordance with literature data to ensure HIC cracking of susceptible steels.[14,15] Test durations shorter than those given in Table 3 for HIC tests in Solution C may not lead to HIC cracking in steels susceptible to HIC under the selected test conditions of Table 3.

NOTE: The test durations given in Table 3 are also appropriate for Fitness-for-Purpose testing in alternate test solutions.

The test time shall begin immediately after saturation with H_2S or the H_2S containing gas mixture is achieved (see Paragraphs 8.2.3 and 8.2.4).

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 metallographically polished, 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 documented preparation procedure that is described in detail and has been proven 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 (2), (3), and (4) respectively and reported for each of the three sections from each test specimen, each individual test specimen as the average of its three sections, and each sample as the overall average of all test specimens.

NOTE: A sample is defined as a set of test specimens.

$$\mathrm{CSR} = \frac{\Sigma\,(a \times b)}{(W \times T)} \times 100\% \tag{2}$$

$$\mathrm{CLR} = \frac{\Sigma\,a}{W} \times 100\% \tag{3}$$

$$\mathrm{CTR} = \frac{\Sigma\,b}{T} \times 100\% \tag{4}$$

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 $(\Sigma a \times \Sigma b)/(W \times T)$, which is simply the product of CLR x CTR (i.e., $\Sigma a/W \times \Sigma b/T$); it does not give the same value as $\Sigma\ (a \times b)/(W \times T)$.

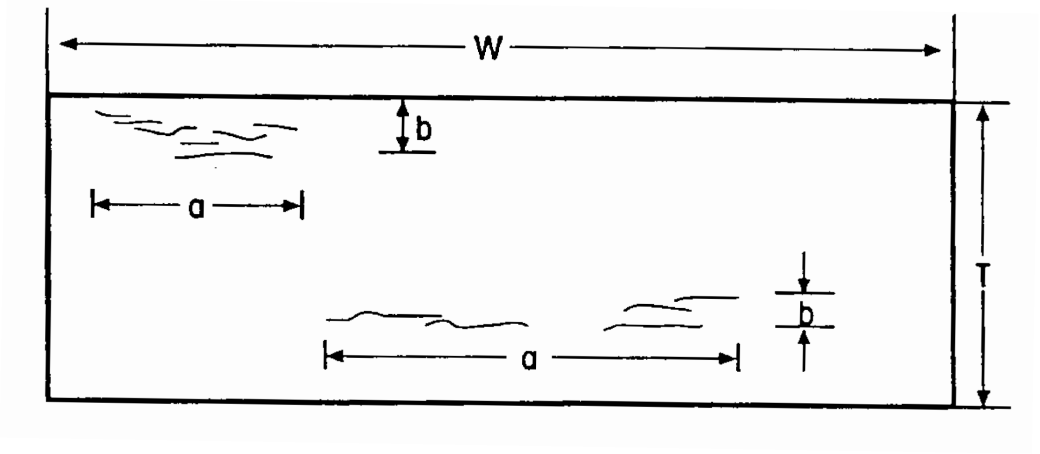

Figure 11: Test Specimen and Crack Dimensions to Be Used in Calculating CSR, CLR, and CTR

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(6) 5L,[16] Grade X52, seamless; ASTM A53,[17] Grade B, ERW; ASTM A516,[18] Grade 70; ASTM A234,[19] Grade WPB; ASTM A105,[20] Grade B; ASTM A350,[21] 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) Location and orientation for each test specimen;

(b) Method of testing for adequacy of test specimen degreasing;

(c) Test solution used (Test Solution A or Test Solution B [Stock Solutions No. 1 and No. 2] or Test Solution C);

(d) Inert purge gas;

(e) Stirring (if applicable) when Test Solution C is used;

(f) pH of the test solution before introduction of H_2S—specified in Paragraphs 8.1.3 and 8.1.4;

(g) pH of the test solution at start of test (after saturation with H_2S or the H_2S containing gas mixture)—specified in Paragraph 8.3.1;

(h) pH of the test solution at end of test—specified in Paragraph 8.3.2;

(i) H_2S content of the test solution at start of test (after saturation with H_2S or the H_2S containing gas mixture)—specified in Paragraphs 8.2.3 and 8.2.4;

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

(j) H_2S content of the test solution at end of test—specified in Paragraphs 8.2.3 and 8.2.4;

(k) Temperature of the test solution—specified in Paragraph 8.5.

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

10.4 The individual CSR, CLR, and CTR values shall be reported for each of the three sections from each test specimen. The average CSR, CLR, and CTR values shall be reported for each individual test specimen as the average of its three sections, and for each sample as the overall average of all test specimens.

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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(8) American Society for Nondestructive Testing (ASNT), PO Box 28518, 1711 Arlingate Lane, Columbus, OH 43228-0518.
(9) British Institute of Non-Destructive Testing (BINDT), Newton Building, St. George's Avenue, Northampton NN2 6JB, U.K.
(10) Aerospace Industries Association of America (AIA), 1250 Eye Street, N.W., Suite 1200, Washington, DC, 20005-3924.
(11) European Committee for Standardization (CEN), Avenue Marnix 17, B-1000 Brussels, Belgium.
(12) Occupational Safety & Health Administration (OSHA), U.S. Department of Labor, 200 Constitution Ave. NW, Washington, DC 20210.
(13) 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 A
Evaluation of Hydrogen Induced Cracking by Ultrasonic Testing
(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.

Section A1: General

A1.1 This procedure details an immersion ultrasonic testing (UT) method to determine the presence, location and area of hydrogen induced cracks in fully machined specimens.

A1.2 The procedure is not applicable to curved specimens or to small fittings tested as complete components.

A1.3 The procedure provides a method for determining the Crack Area Ratio (CAR).

Section A2: Terms and Definitions

A-Scan Display: A data presentation method in which signal amplitude is plotted along the y-axis versus time on the x-axis. The horizontal distance between any two signals represents the material distance between the two reflectors causing the signals. In a linear system, the vertical excursion is proportional to the amplitude of the signal.

B-Scan Display: Refers to the image produced when the data collected from an ultrasonic inspection is plotted on a cross-sectional view of the specimen.

C-Scan Display: Refers to the image produced when the data collected from an ultrasonic inspection is plotted on a plan view of the specimen.

D-Scan Display: Refers to the image produced when the data collected from an ultrasonic inspection is plotted on an end view of the specimen.

DAC: Distance **A**mplitude **C**orrection**:** is a method of compensating for the fact that the pulse-echo response of a reflector will decrease as the distance of the reflector from the ultrasonic probe increases. This occurs because the transmitted ultrasonic beam spreads out as it travels from the probe to the reflector and so the further the reflector is from the probe the lower the energy of the sound that actually hits the reflector. Similarly, the further the reflected pulse has to travel the lower the energy that is received back at the probe.

TCG: Time-**C**orrected **G**ain is a method of compensating for a reduction in signal amplitude with increasing range from reflectors of equal area. This is achieved by increasing the system gain with time so that the signals appear of equal amplitude. TCG achieves the same objective as a DAC.

Time of Flight: The time for an acoustic wave to travel between two points. For example, the time required for a pulse to travel from the transmitter to the receiver via diffraction at a discontinuity edge or along the surface of the test object.

Section A3: Qualification of Personnel

A3.1 The equipment shall be set up and the work supervised by personnel qualified in a recognized certification scheme to the appropriate level. Examples of certification schemes are shown below:

i) American Society for Nondestructive Testing (SNT-TC-1A/ASNT[22])
ii) Personnel Certification for Non-destructive Testing (PCN)[23]
iii) NAS 410: Certification & Qualification of NDT Personnel[24]
iv) ISO 9712: Qualification and Certification of NDT Personnel[25]
v) EN 4179: Certification & Qualification of NDT Personnel[26]

Section A4: Ultrasonic Test Method

A4.1 The ultrasonic test method shall be fully or semi-automatic using the pulse-echo method and compression waves.

A4.2 The method shall use the immersion technique with clean degassed water as the coupling medium. Corrosion inhibitors may be added if required. The calibration of sensitivity shall be performed in the degassed coupling medium with inhibitor (if added).

Section A5: Equipment

A5.1 Ultrasonic Generator (instrument)

A5.1.1 The ultrasonic generator shall be subjected to an annual calibration. A calibration certificate for the unit shall be available detailing calibration results traceable to the relevant National Standard.

A5.1.2 The ultrasonic generator shall be capable of being adjusted using one of these two methods:

i) Distance Amplitude Correction (DAC).
The threshold of the monitor varies over the depth (time-of-flight).
ii) Time Corrected Gain (TCG).
The gain of the receiver depends on the depth (time-of-flight).

A5.1.3 In both cases, several artificial reflectors with unique shapes but different depths are used for adjustment.

A5.2 Transducer

A5.2.1 The transducers (probes) used may be of the single element or double element type (transmit/receive [TR]) and shall be compatible with the ultrasonic generator used.

A5.2.2 The ultrasonic transducer shall be subjected to an annual calibration. A calibration certificate for the transducer shall be available detailing calibration results traceable to the relevant National Standard.

A5.2.3 The transducers shall have the following characteristics:

i) Circular, flat or cylindrically focused transducer.
ii) Up to 20 mm in diameter. The transducer diameter shall not exceed the width of the specimen.
iii) Frequency = 10 – 25 MHz

Section A6: Specimen Preparation

A6.1 The scanning surfaces shall be free from corrosion products, scale or any other foreign material that may prevent adequate transmission of the sound waves across the water-specimen interface.

Section A7: Calibration

A7.1 Calibration of Time Base

A7.1.1 The calibration of the time base shall be carried out on a calibration block manufactured from an undisturbed area of one of the samples to be tested or from a material with an equivalent grade, condition, and acoustic properties.

A7.1.2 The monitor gate for measuring echoes from indications shall be adjusted as follows:

i) Start of gate: immediately after incidence echo or in the case of the double type (TR) probe at 0 mm depth.
ii) End of gate: just before the first back wall echo.

A7.1.3 The time-of-flight shall be measured between the water/specimen interface echo and the first back wall echo, or between the first and second back wall echoes. This value shall be used to calibrate the Time Base and/or the sound velocity for thickness or depth measurements in order to obtain same result of mechanical measuring and ultrasonic measurements.

A7.2 Calibration of Sensitivity

A7.2.1 The aim of DAC or TCG is to obtain uniform test sensitivity independent from the depth of an indication. The variation of the test sensitivity depends mainly on the transducer used. Therefore, it is not necessary to manufacture calibration blocks for each dimension of samples to be tested. Differences in sound attenuation given by different grades and conditions can be compensated for by comparison of back wall echoes in undisturbed areas of the specimen with back wall echoes from areas of the calibration block while both samples are turned to 90° (incidence in the unique width of the samples).

A7.2.2 The calibration block shall have the dimensions of 20 mm width, 100 mm length and a wall thickness greater than or equal to the wall thickness of the specimen to be tested.

A7.2.3 The thickness of the calibration block shall be either:

i) the same as the specimen to be tested.
ii) one of the thicknesses of t = 10 mm, t = 20 mm, t = 30 mm and t = 40 mm

A7.2.4 In case of using a calibration block with the same thickness as the specimen to be tested or a calibration block with 10 mm or 20 mm thickness, the block shall contain five transverse flat bottom slots, 1 mm in width, at depths of 1 mm, 1/4t, 1/2t, 3/4t and t less 1 mm, as shown in Figure A1.

A7.2.5 In case of using the calibration block of 30 mm thickness, the block shall contain seven transverse flat bottom slots, 1 mm in width, at depths of 1 mm, 5 mm, 10 mm, 15 mm, 20 mm, 25 mm and 29 mm, as shown in Figure A2.

A7.2.6 In case of using the calibration block of 40 mm thickness, the block shall contain 9 transverse flat bottom slots, 1 mm in width, at depth of 1 mm, 5 mm, 10 mm, 15 mm, 20 mm, 25 mm, 30 mm, 35 mm and 39 mm, as shown in Figure A3.

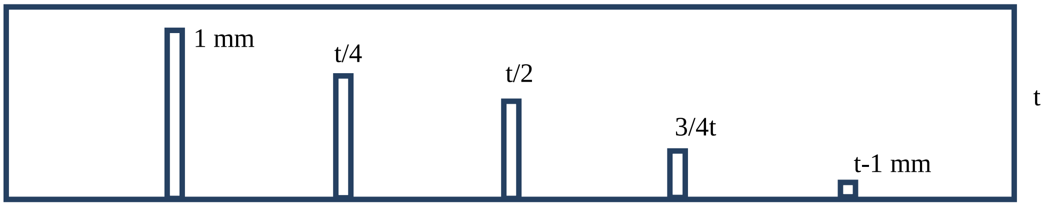

Figure A1: Location of Machined Slots in Calibration Block (Side View)

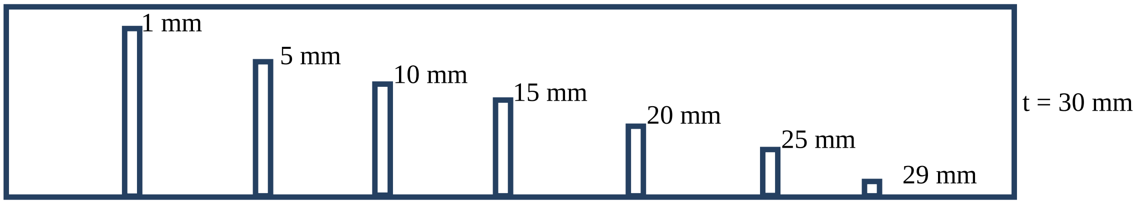

Figure A2: Location of Machined Slots in Calibration Block (Side View)

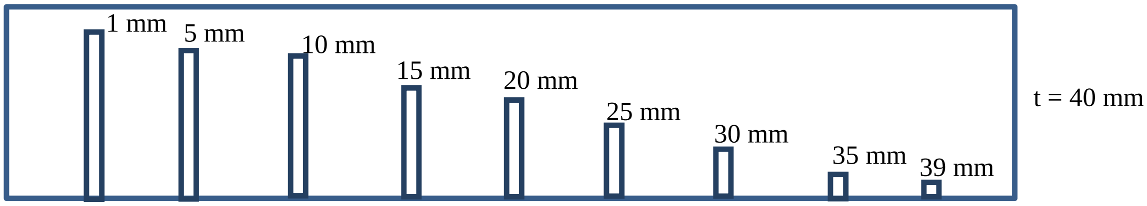

Figure A3: Location of Machined Slots in Calibration Block (Side View)

A7.2.7 The sensitivity may be determined using either the DAC curve or the TCG level methods.

A7.2.8 The echo from the 1 mm deep notch shall be adjusted to 80% Full Screen Height (FSH) for both the DAC curve and the TCG methods.

A7.2.9 The threshold / reference level shall be either the DAC curve or the TCG level. All indications equal to or above the DAC curve or the TCG level (whichever is applicable) shall be recorded.

A7.2.10 It shall be shown that the echo resulting upon reflection at the deepest notch (t -1 mm) is clearly distinguishable from the back wall echo.

A7.2.11 By turning the samples by 90° (incidence from the side), the first back wall echoes from the calibration block and the specimen to be tested shall be compared. Differences shall be compensated for.

A7.3 Transducer Location & Movement

A7.3.1 The distance between the transducer and the specimen surface (water path length) shall be accurately adjusted and recorded. For all measurements (calibration and testing), the same water path length shall be compared.

A7.3.2 The transducer shall move parallel to the surface within ±0.5° in all directions.

A7.3.3 The transducer movement shall be smooth without any stopping.

A7.3.4 The movement speed shall be less than 130 mm/second.

A7.3.5 The scanning pitch is guaranteed to ensure an overlapping of a minimum of 20% of the transduced diameter, since the probe diameter is specified from 10 to 20 mm and the scan resolution is specified as 0.5 mm or finer.

A7.4 Extent of UT Inspection

A7.4.1 Scanning shall cover 100% of the required specimen surface. During scanning, the beam axis of the probe shall be moved from upper to lower edge. The first scan shall be carried out while the beam axis travels along the left edge and the last scan shall be carried out with the beam axis on the right edge.

A7.4.2 The scan resolution shall be 0.5 mm x 0.5 mm or finer.

A7.4.3 The ultrasonic beam shall be oriented perpendicular to the test surface.

A7.4.4 For parent pipe and plate specimens, the scanned face shall either be the outer or inner faces of the specimen.

A7.4.5 For specimens machined from the electric resistance weld (ERW) region of welded pipes, scanning of the one side face, in addition to the outer or inner faces, shall also be undertaken due to the possibility of the indication (HIC) orientation running in the through thickness direction. The CAR for side face scanning can be calculated in accordance with Section A8.

A7.4.6 For specimens machined from welds, forged or cast products scanning of the one side face, in addition to the outer or inner faces, shall also be undertaken due to the possibility of the indication (HIC) orientation running in the through thickness direction. The CAR for side face scanning can be calculated in accordance with Section A8.

NOTE: Scanning of the one side face before and after the HIC test may help distinguish between HIC cracks and other sources of UT indications.

A7.5 Cartography / Printouts

A7.5.1 A C-scan chart shall be produced.

A7.5.2 Optional A-scan or B-scan charts may also be produced for information.

A7.5.3 The charts / graphs produced shall give the size and location of detected indications to an accuracy of ± 0.5 mm or finer.

A7.5.4 The amplitude of echoes from cracks may be referenced in different colors.

Section A8: Crack Area Ratio (CAR)

A8.1 The CAR shall be determined for each specimen and calculated as follows:

CAR % = Total Area of Indications x 100 (D1) / Area of specimen surface*

* The area of the specimen surface is the surface area of the scanned face only.

or:

CAR % = total number of measuring points above threshold x 100 / total number of measuring points of the specimen.

A8.2 For side face scanning, the CAR shall also be determined and reported separately to the outer / inner face results. This shall be identified on the test report and taken into account when applying any acceptance criteria.

Section A9: Reporting

A9.1 The following minimum information shall be reported:

A9.1.1 The transducer type (i.e. single or double), diameter and frequency.

A9.1.2 Charts of the C-scan indications (if any).

A9.1.3 The CAR percentage for each specimen.

Appendix B
Safety Considerations in Handling H_2S Toxicity
(Nonmandatory)

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

H_2S is perhaps responsible for more industrial poisoning accidents than is any other single chemical. A number of these accidents have been fatal. H_2S must be handled with caution and any experiments using it must be planned carefully. The OSHA maximum allowable concentration of H_2S in the air for an eight-hour work day is 20 parts per million (ppm), well above the level detectable by smell.[27] However, the olfactory nerves can become deadened to the odor after exposure of 2 to 15 minutes, depending on concentration, so that odor is not a reliable alarm system.

Briefly, the following are some of the human physiological reactions to various concentrations of H_2S. Exposure to concentrations in the range of 150 to 200 ppm for prolonged periods may cause edema of the lungs. Nausea, stomach distress, belching, coughing, headache, dizziness, and blistering are 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 H_2S can be obtained from the Chemical Safety Data Sheet SD-36 [28] and from *Dangerous Properties of Industrial Materials*.[29]

Fire and Explosion Hazards

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

Safety Procedures During Test

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

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

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

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

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

Test Equipment

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

Test Reagents

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

Analytical grade reagents shall be used.

Titration Procedure

D1 Table D1 gives the titration parameters required for determination of the test solution H_2S concentration, for a series of H_2S partial pressure ranges from 1.00 bar to less than 0.01 bar. The limits for each range in the series have been chosen to ensure that analytical accuracy is maintained. The range selected shall be appropriate to the test H_2S partial pressure.

Table D1
Titration Parameters for Determination of the Test Solution H_2S Concentration.

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

D2 Pipette 25 mL of the selected (0.1 N or 0.01 N) standard iodine solution into a conical flask.

D3 Acidify with a few drops of concentrated HCl. If sampling using a syringe (see Paragraph D4.3), weigh the conical flask containing the acidified iodine solution and record the result.

D4.1 When sampling using a volumetric pipette, transfer an initial 25-50 mL of test solution from the test vessel into a clean beaker. Rinse the beaker with the test solution and discard. Transfer a further 50-100 mL of test solution to the beaker. Fill the pipette from the beaker, rinse with the test solution and discard. Extract the required volume of test solution from the beaker via the pipette, and immediately transfer to the conical flask containing the acidified iodine solution. Record the volume of test solution transferred.

D4.2 When sampling using a volumetric flask, transfer an initial 25-50 mL of test solution from the test vessel into the flask. Rinse the flask with the test solution and discard. Transfer sufficient test solution to fill the flask to the required volume, and immediately transfer to the conical flask containing the acidified iodine solution. Record the volume of test solution transferred.

D4.3 When sampling using a syringe, extract an initial 25-50 mL of test solution from the test vessel. Rinse the syringe with the test solution and discard. Refer to the graduations on the syringe to extract the approximate volume of test solution required, and transfer directly to the pre-weighed conical flask containing the acidified iodine solution. Re-weigh the conical flask and record the result. Determine the exact volume of test solution transferred.

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

D5 Titrate with the selected (0.1 N or 0.01 N) standard sodium thiosulfate solution until the solution changes color from dark yellow/tan to pale yellow.

D6 Add a few drops of starch solution.

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

D7 Continue to titrate slowly with the selected (0.1 N or 0.01 N) standard sodium thiosulfate solution until the dark blue color disappears, and the end point is reached. The end point is a milky yellow suspension of colloidal sulfur. Record the total volume of standard sodium thiosulfate solution used.

D8 Calculate the H_2S concentration (mg/L) using Equation (D1):

$$\mathrm{H_2S\ concentration\ (mg/L)} = \frac{(\mathrm{A} - \mathrm{B})}{\mathrm{C}}\ \mathrm{x}\ 17{,}040 \qquad \text{(D1)}$$

where:

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

$$17{,}040 = \frac{34.08\ \mathrm{(g/mole\ H_2S)\ x\ 1000\ (mg/g)}}{2\ \mathrm{(equivalents/mole\ H_2S)}}$$

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