



Designation: G186 – 05

Standard Test Method for Determining Whether Gas-Leak-Detector Fluid Solutions Can Cause Stress Corrosion Cracking of Brass Alloys¹

This standard is issued under the fixed designation G186; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers an accelerated test method for evaluating the tendency of gas leak detection fluids (LDFs) to cause stress corrosion cracking (SCC) of brass components in compressed gas service.

1.2 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*²

B135 Specification for Seamless Brass Tube

B135M Specification for Seamless Brass Tube [Metric]

D1193 Specification for Reagent Water

G1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens

G15 Terminology Relating to Corrosion and Corrosion Testing

G37 Practice for Use of Mattsson's Solution of pH to Evaluate the Stress-Corrosion Cracking Susceptibility of Copper-Zinc Alloys

G38 Practice for Making and Using C-Ring Stress-Corrosion Test Specimens

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

¹ This test method is under the jurisdiction of ASTM Committee G01 on Corrosion of Metals, and is the direct responsibility of Subcommittee G01.06 on Environmentally Assisted Cracking.

Current edition approved July 1, 2005. Published July 2005. DOI: 10.1520/G0186-05.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.1.1 *Gas Leak Detector Solutions*—Also known as leak detection fluids, leak detector solutions, bubble solutions, and soap solutions, designated in this standard as LDFs, are fluids used to detect leaks in pressurized gas systems by the formation of bubbles at the leak site.

3.1.2 The terminology used herein, if not specifically defined otherwise, shall be in accordance with Terminology G15.

4. Summary of Test Method

4.1 This test method consists of three steps: The first step consists of running a sample of the test specimens to verify that they are susceptible to stress corrosion cracking using Mattsson's Solution (see Practice G37). The second step is to expose the specimens to a solution that does not cause SCC to verify that the test environment does not contain components that can cause SCC to brass. The third step is to test the LDF to determine if it causes SCC of the brass specimens within 15 wetting and evaporation cycles.

4.2 The specimen used in this test is a C-ring stressed to create at least 0.65% strain in the outer fibers of the specimen.

4.3 Macroscopic examination of the specimens is carried out after every second wetting cycle and if cracking is suspected the specimen is examined at higher magnifications for confirmation. Metallographic sectioning through the stressed area is used to verify minor cracking at the end of the fifteen cycles.

4.4 LDFs that cause SCC in any specimens within 15 wetting cycles are considered to have failed this test and not suitable for use in pressurized gas systems with brass components.

5. Significance and Use

5.1 Brass components are routinely used in compressed gas service for valves, pressure regulators, connectors and many other components. Although soft brass is not susceptible to ammonia SCC, work-hardened brass is susceptible if its hardness exceeds about 54 HR 30T (55HRB) (Rockwell scale). Normal assembly of brass components should not induce sufficient work hardening to cause susceptibility to ammonia SCC. However, it has been observed that over-tightening of the components will render them susceptible to SCC, and the problem becomes more severe in older components that have

been tightened many times. In this test, the specimens are obtained in the hardened condition and are strained beyond the elastic limit to accelerate the tendency towards SCC.

5.2 It is normal practice to use LDFs to check pressurized systems to assure that leaking is not occurring. LDFs are usually aqueous solutions containing surfactants that will form bubbles at the site of a leak. If the LDF contains ammonia or other agent that can cause SCC in brass, serious damage can occur to the system that will compromise its safety and integrity.

5.3 It is important to test LDFs to assure that they do not cause SCC of brass and to assure that the use of these products does not compromise the integrity of the pressure containing system.

5.4 It has been found that corrosion of brass is necessary before SCC can occur. The reason for this is that the corrosion process results in cupric and cuprous ions accumulating in the electrolyte. Therefore, adding copper metal and cuprous oxide (Cu_2O) to the aqueous solution accelerates the SCC process if agents that cause SCC are present. However, adding these components to a solution that does not cause SCC will not make stressed brass crack.

5.5 Repeated application of the solution to the specimen followed by a drying period causes the components in the solution to concentrate thereby further increasing the rate of cracking. This also simulates service where a system may be tested many times during its life. These features of the test method accelerate the test and allow an answer to be obtained more rapidly.

5.6 This test method applies only to brasses. Successful passage of this test does not assure that the LDF will be acceptable for use on other alloy systems such as stainless steels or aluminum alloys.

6. Interferences

6.1 When conducting this test, it is very important that the air not be contaminated with ammonia vapors. Reagent bottles with ammonium hydroxide or other tests that involve ammonia or its compounds including amines must not be in the vicinity of these tests. This also includes Mattsson's test solution.

6.2 Cross contamination may result in false stress corrosion cracking results therefore concurrent exposure tests with different leak detector solutions should be conducted in such a way that any set of samples does not influence the results of any of the other samples.

6.3 In this test, the susceptibility of the C-ring specimens to crack in a particular test solution can be affected by the temper of the brass alloy; therefore, it is crucial that the C-rings be fabricated from hard drawn temper brass tubing that meets the minimum specified hardness requirements.

7. Reagents and Materials

7.1 Reagent grade cuprous oxide (Cu_2O) and USP/FCC grade glycerin ($\text{C}_3\text{H}_8\text{O}_3$) conforming to specifications of the

committee on Analytical Reagents of the American Chemical Society, where such specifications are applicable,³ shall be used.

7.2 Fine pure copper powder with particle size $<68\ \mu\text{m}$ shall be used.

7.3 Solutions using water shall be prepared using distilled or deionized water conforming to the purity requirements of Specification **D1193**, Type IV reagent water.

7.4 Leak detector solutions shall meet manufacturer's specifications.

7.5 Mattsson's Solution shall be freshly prepared according to Practice **G37**.

8. Hazards

8.1 Consult Material Safety Data Sheets (MSDS) for all chemicals both reagent and commercial before testing to gain a full understanding of any potential hazards.

8.2 The test solutions present no undue safety hazard. It is recommended, however, that appropriate personnel protection equipment such as resistant gloves and shatterproof eyewear with side shields be worn when the chemicals or specimens are handled.

8.3 The solutions contain copper and are thus considered poisonous so they should not be ingested.

8.4 Ammonium sulfate, $(\text{NH}_4)_2\text{SO}_4$, in the Mattsson's solution has been reported to be allergenic. Repeated short-time skin contact with the solution over extended periods of time should be avoided.

8.5 The fumes given off by the Mattsson's test solution contain ammonia. The least detectable ammonia odor corresponds to a concentration of 50 ppm; 100 ppm can be tolerated for several hours without serious disturbance; 700 ppm causes immediate eye irritation; and greater than 5000 ppm can be lethal. The mixing and the actual testing with Mattsson's solution should therefore be run in a well-ventilated area.

9. Test Solutions

9.1 *Control Solution (benign water solution)*—Add 2.5 g Cu powder and 2.5 g Cu_2O to 1000 ml of H_2O . Then add 10ml of glycerin to solution. Shake solution vigorously to thoroughly mix contents.

9.2 *Leak Detector Solutions*—Add 2.5 g Cu powder and 2.5 g Cu_2O to 1000 ml of each manufacturer's solution to be tested. Shake solutions vigorously to thoroughly mix contents.

NOTE 1—Some of the solids will settle out of the solutions in between cycles, therefore, it is very important to shake the solutions vigorously prior to their use in the wetting cycle.

³ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

10. Test Specimen

10.1 *Type and Size*—An unnotched C-ring in accordance with Practice G38 shall be used (Fig. 1). C-rings shall have an outer diameter (OD) between 1.0 to 2.0 in. (25.4 to 50.8 mm) and thickness between 0.065 to 0.25 in. (1.65 to 6.4 mm). Widths shall be fixed at 0.75 in. (19.0 mm).

10.2 *Alloy Composition and Temper*—Test specimens shall be fabricated from copper alloy (UNS C27200) seamless tube with a H80 hard-drawn temper. The hard drawn temper shall have a minimum hardness of 70 on the 30T Rockwell scale which corresponds to minimum tensile strength of 66 ksi (455 MPa). Refer to Specifications B135 and B135M for tube information.

10.3 *Surface Finish*—Specimens shall have a 120-grit finish. Grind marks shall be in the circumferential direction.

10.4 *Restraining hardware*—Nuts, bolts, and flat washers shall be made from a metal resistant to the chemicals used in this test (for example, Type 316 stainless steel, UNS S31600). Insulation washers shall be made from a hard resistant material to prevent relaxation of the stressed C-ring specimen (for example, alumina). Refer to Fig. 1.

10.5 *Pre-test Condition*—C-rings will be un-stressed prior to testing.

10.6 *Stressing Method*—Constant-strain stressing method shall be used in stressing the C-rings (refer to Practice G38). Tensile stress will be introduced on the exterior of the ring by tightening a bolt centered on the diameter of the ring.

10.7 *Surface Preparation*—The specimen surface should be free of oil, grease, and dirt. This usually entails cleaning with organic solvent such as acetone followed by an alcohol rinse.

NOTE 2—Every precaution shall be taken to maintain the integrity of the surface after the final preparation. Avoid rough handling that could mar

the surface and handle with gloves to prevent fingerprinting and the transfer of contaminants.

11. Test Setup and Apparatus

11.1 *C-ring Test Assembly*—Individual C-ring specimens shall be placed apex down in a glass-fiber-wick covered dish (Fig. 3)

11.2 *Exposure Dish*—The individual dishes shall be made out of a material that will not react with the chemicals being used for the exposure (for example, glass, polystyrene, polycarbonate). The required dish dimensions are 2.36- to 3.94-in. (60- to 100-mm) diameter and 0.59- to 0.79-in. (15- to 20-mm) height.

11.3 *Wick Material*—Borosilicate glass wick material with a fiber diameter of about 0.3 mil (8 μm) shall be used.

11.4 *Number of Specimens*—Five individual C-ring specimen test assemblies will be used for each leak detector or control solution to be tested.

11.5 *Test Assembly Arrangement*—Individual exposure dishes shall be laid out such that there is at least a 1.0 ft (305 mm) separation between groups of specimens (Fig. 4). The control solution specimens shall always be positioned in the center of the test group.

12. Calibration and Standardization

12.1 When a new batch of specimens is to be used, it is necessary to first test a representative number of C-rings from the batch with Mattsson’s solution.

12.2 Mattsson’s Solution test must produce cracking of the test specimens before any further testing is carried out.

12.3 If cracking is not produced during the prescribed Mattsson’s solution test the following variables need to be verified before rejecting the batch of C-rings.

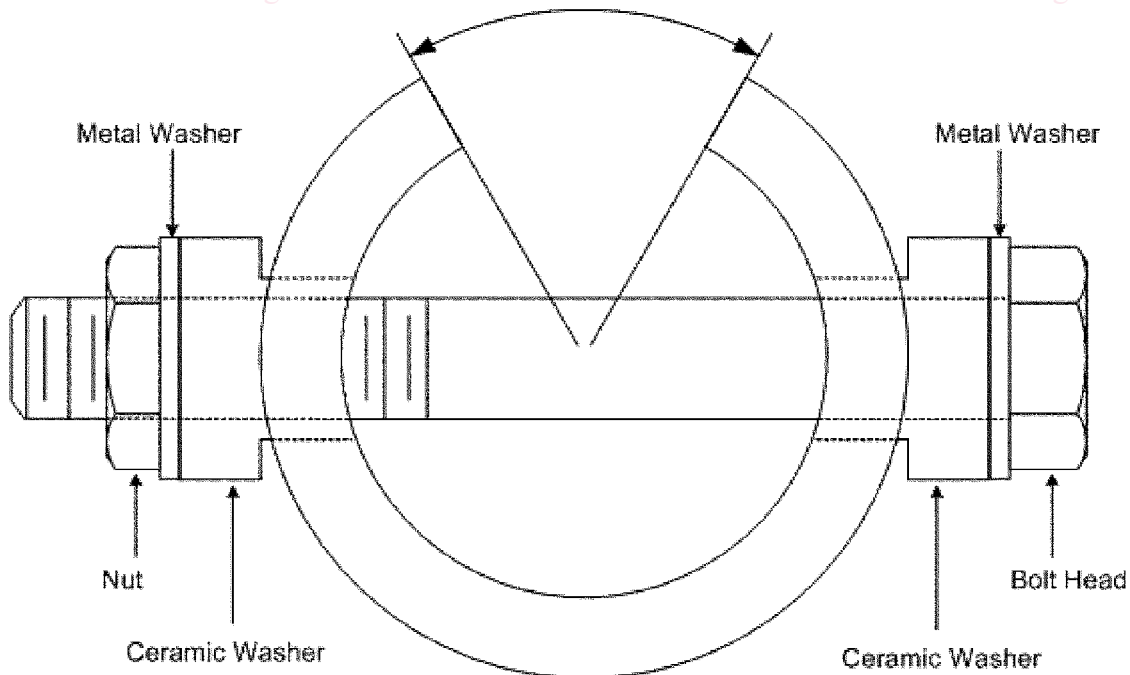


FIG. 1 C-ring Specimen

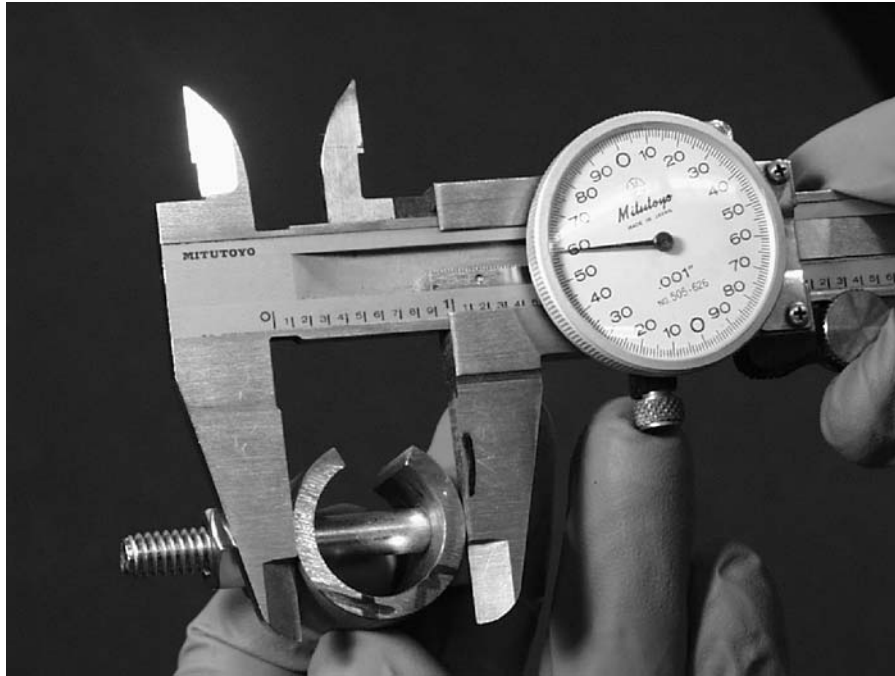


FIG. 2 Caliper Measurement of C-ring's Deflection

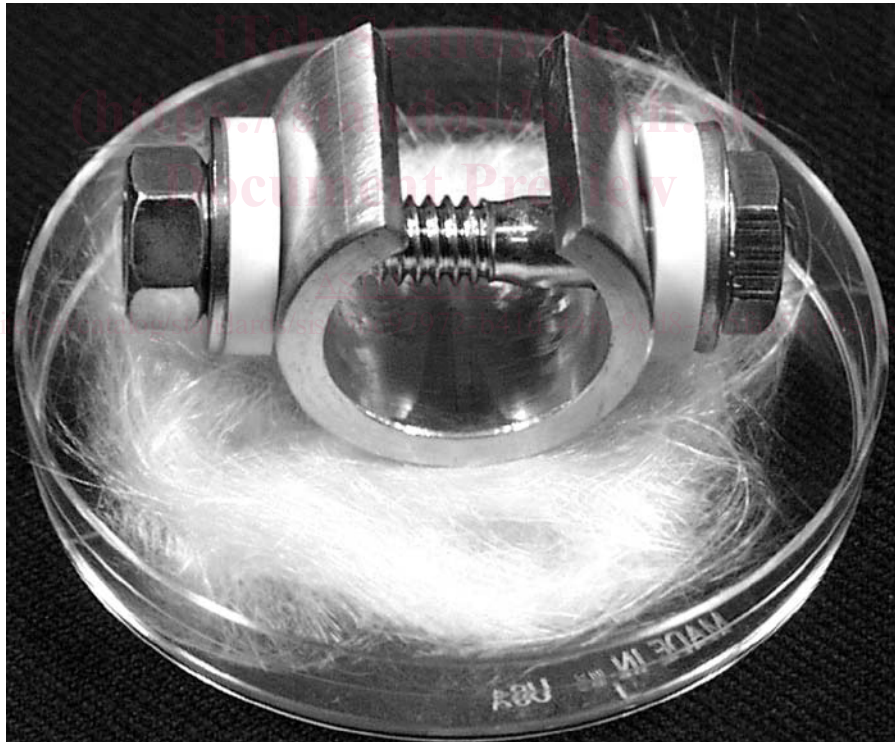


FIG. 3 C-ring Specimen Ready for Exposure to Test Solution

12.3.1 Specimen material hardness shall exceed 70 HR 30T (Rockwell scale).

12.3.2 Strain should be checked with a strain gauge if hardness is sufficient.

12.3.3 Mattsson's solution should be checked to make sure it conforms to Practice G37.

12.4 Follow steps in Annex Section Annex A1 to test the C-rings in Mattsson's Solution.

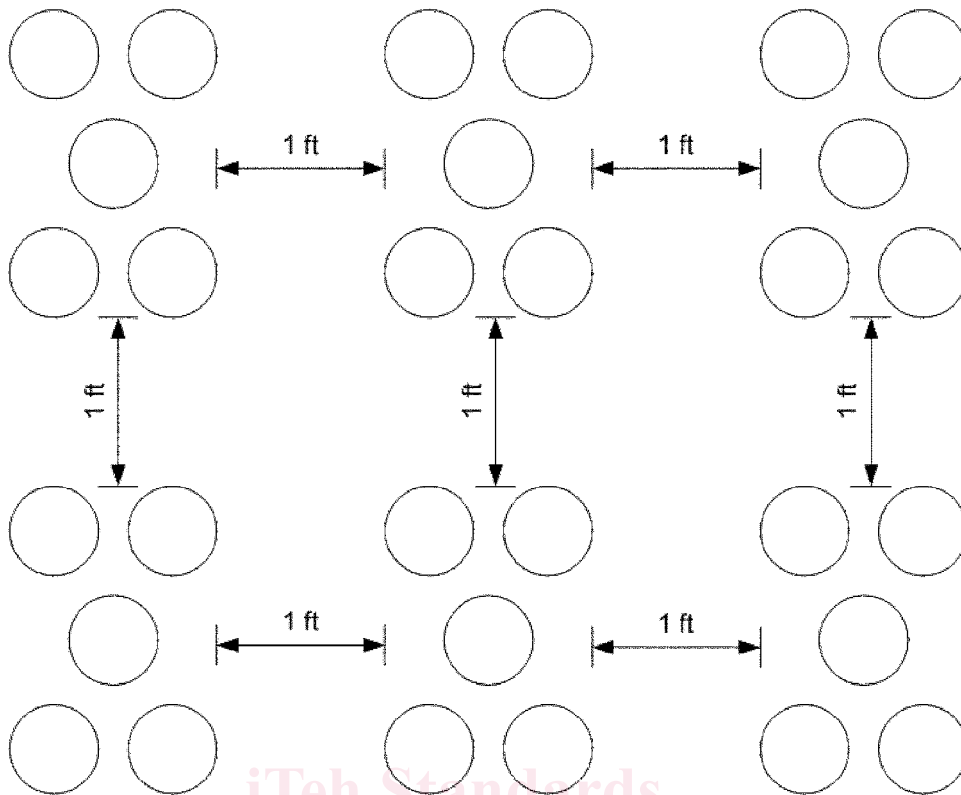


FIG. 4 Arrangement and Spacing of Specimens for Multiple Solution Testing

13. Air Conditions

13.1 *Temperature*—Air temperature shall be maintained in a range of 70 – 80°F (21 – 27°C) throughout the entire test duration.

13.2 *Relative Humidity*—Percent relative humidity of the air shall be maintained in a range of 15 to 60 % throughout the entire test cycle.

13.3 Air Circulation

13.3.1 Air circulation is considered to be very important to testing since it affects the rate at which the solutions lose water by evaporation. Optimum conditions for air circulation have not been established, but recommendations described in 13.3.2 shall be considered.

13.3.2 The most important consideration is to achieve moderate evaporation of the test solutions in a 2-3 day time period.

13.3.3 Stagnant air conditions should be avoided.

13.3.4 Testing in a ventilated enclosure may be required to achieve adequate circulation.

NOTE 3—Care should be taken to avoid overnight and weekend changes in operation of the laboratory heating, ventilation, and air conditioning equipment which could result in systematic excursions outside the desired temperature, humidity, and air circulation ranges.

14. Procedure

14.1 Leak detector fluid and control solutions are tested concurrently. No Mattsson’s solution tests shall be conducted while control or test solutions are in progress.

14.2 Stressing of the C-rings:

14.2.1 The specimens shall be stressed the same way for exposures in the leak detector fluid and control solutions.

14.2.2 Individual specimens should be assembled as shown in Fig. 1.

14.2.3 Constant strain stressing method shall be used in stressing the C-rings. Tensile stress will be introduced on the exterior of the ring by tightening the bolt centered on the diameter of the ring.

14.2.4 Tighten the restraining bolt and nut until the deflection required to produce a total strain of 0.65% on the outer fiber of the C-ring specimen is achieved.

14.2.5 The required deflection can be determined from Table 1, which is based on the outer diameter and wall thickness of the original unstressed C-ring or the formula given in Appendix X1.

14.2.6 Measure the deflection of the C-ring across the faces perpendicular to the bolt as seen in Fig. 2.

TABLE 1 Deflection for Stressing C-rings

O.D. (inch)	Wall Thickness (in)				
	0.065	0.08	0.091	0.125	0.25
1	0.073	0.059	0.051	0.036	0.015
1-1/16	0.083	0.067	0.058	0.041	0.018
1-1/8	0.094	0.075	0.065	0.046	0.020
1-3/16	0.105	0.089	0.073	0.052	0.023
1-1/4	0.116	0.083	0.081	0.057	0.026
1-5/16	0.129	0.099	0.090	0.064	0.028
1-3/8	0.142	0.114	0.099	0.070	0.032
1-1/2	0.169	0.136	0.119	0.084	0.038
1-9/16	0.184	0.148	0.129	0.092	0.042
1-5/8	0.199	0.160	0.140	0.100	0.046
1-3/4	0.232	0.187	0.163	0.116	0.054
1-7/8	0.267	0.215	0.188	0.134	0.062
2	0.304	0.245	0.214	0.153	0.072

14.3 *Assembly of Individual Exposure Dishes:*

14.3.1 Make sure individual dishes are clean of any foreign matter.

14.3.2 Cut swatches of glass fiber wick about 1/8 in. (3 mm) thick to fit the bottom of the dishes.

14.3.3 Place the layer of glass fiber wick across the bottom of the individual dishes.

14.3.4 Place the C-rings inverted into the individual dishes. The apex of the C-ring should be resting on the wick material at the bottom of the dish (Fig. 3).

14.3.5 The exposure dishes shall be laid out in such a pattern that there is at least 1 ft (305 mm) separation between groups of specimens (Fig. 4). The control solution specimens shall be located at the center of the exposure dish cluster.

14.4 *Exposing the Specimens:*

14.4.1 Start exposure by temporarily removing the individual C-ring specimens and add 10 ml of leak detector fluid and control solution to corresponding exposure dish.

14.4.2 Replenish LDF solutions (wetting cycle) in each of the corresponding exposure dish by adding an additional 10 ml every 2-3 days (evaporation cycle) (Fig. 5).

14.4.3 Continue to add the leak detector and control solutions for a total of 15 cycles (including initial addition) or until specimen cracks.

14.4.4 At the end of the 15 cycles examine the individual C-rings for cracks. If no cracks are detected continue exposure of the C-ring for an additional 7 days with no further test solution additions.

14.5 *Inspection of Specimens (during exposure):*

14.5.1 Specimens shall be visually inspected at least every 2 cycles prior to the next solution addition.

14.5.2 C-rings should be removed from their test assembly for visual inspection. A low magnification up to 50× may be required to identify fine cracks.

14.5.3 Do not remove dried solutions from surface of C-rings. If surface cannot be seen through dried solution films, continue testing until an obvious crack is present or test duration is reached.

14.5.4 If cracks are detected or the test duration is reached, testing is finished for that particular C-ring specimen.

14.6 *Cleaning of Specimens (after testing):*

14.6.1 Upon discovery of crack or at completion of testing, rinse the specimens thoroughly with distilled or deionized water.

14.6.2 Disassemble rinsed specimens.

14.6.3 Remove any remaining dried leak detector fluid solution with a soft bristle brush and water rinse.

14.6.4 Dry specimen after cleaning/rinsing.

14.6.5 Remove corrosion products if needed with acid cleaning as outlined in Practice G1. Use acid mixtures according to designations C.2.3 and C.2.4.

15. **Final Examination of Specimens**

15.1 *Macroscopic Examination:*

15.1.1 Perform examination at a magnification of at least 25× on all specimens after cleaning. Examine the entire bend section of the specimen. Stress corrosion cracking does not always occur at the apex.

15.1.2 The cleaned C-rings may need to be re-stressed in order to clearly detect any cracks.

15.2 *Microstructural Examination:*

15.2.1 Perform a microscopic examination on any specimen, if the presence of SCC is questionable.

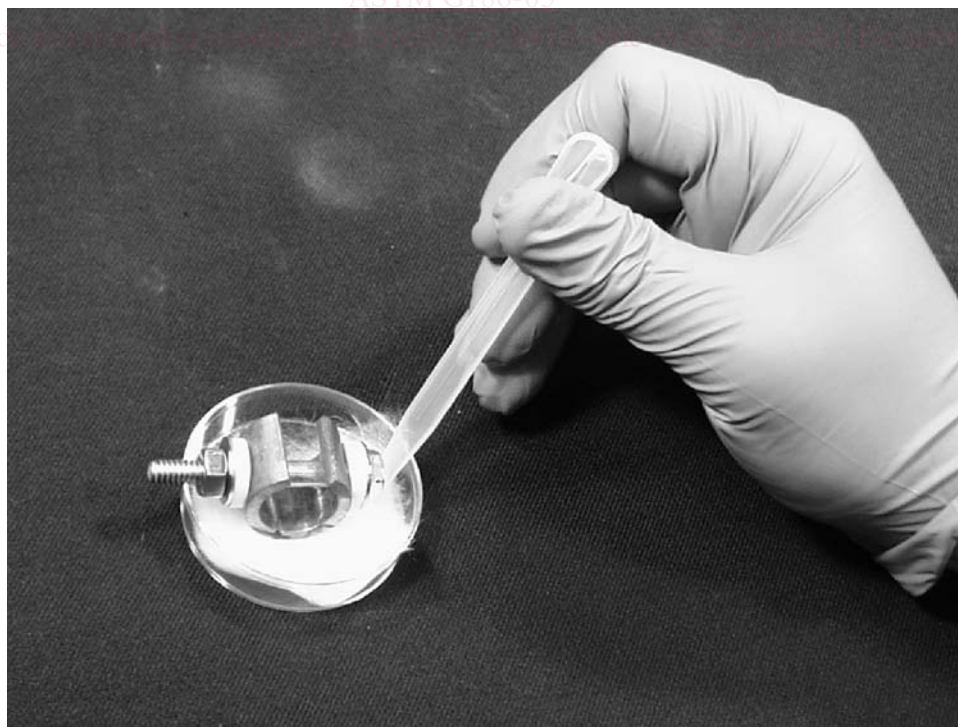


FIG. 5 Replenishment of Test Solution to Individual C-ring