Designation: C1617 − 19

Standard Practice for Quantitative Accelerated Laboratory Evaluation of Extraction Solutions Containing Ions Leached from Thermal Insulation on Aqueous Corrosion of Metals[1]

This standard is issued under the fixed designation C1617; 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 practice covers procedures for a quantitative accelerated laboratory evaluation of the influence of extraction solutions containing ions leached from thermal insulation on the aqueous corrosion of metals. The primary intent of the practice is for use with thermal insulation and associated materials that contribute to, or alternatively inhibit, the aqueous corrosion of different types and grades of metals due to soluble ions that are leached by water from within the insulation. The quantitative evaluation criteria are Mass Loss Corrosion Rate (MLCR) expressed in mils per year determined from the weight loss due to corrosion of exposed metal coupons after they are cleaned.

1.2 This practice cannot cover all possible field conditions that contribute to aqueous corrosion. The intent is to provide an accelerated means to obtain a non-subjective numeric value for judging the potential contribution to the corrosion of metals that can come from ions contained in thermal insulation materials or other experimental solutions. The calculated numeric value is the mass loss corrosion rate. This calculation is based on general corrosion spread equally over the test duration and the exposed area of the experimental cells created for the test. Corrosion found in field situations and this accelerated test also involves pitting and edge effects and the rate changes over time.

1.3 The insulation extraction solutions prepared for use in the test can be altered by the addition of corrosive ions to the solutions to simulate contamination from an external source. Ions expected to provide corrosion inhibition can be added to investigate their inhibitory effect.

1.4 Prepared laboratory ionic solutions are used as reference solutions and controls, to provide a means of calibration and comparison.[2]

1.5 Other liquids can be tested for their potential corrosiveness including cooling tower water, boiler feed, and chemical stocks. Added chemical inhibitors or protective coatings applied to the metal can also be evaluated using the general guidelines of the practice.

1.6 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.

1.7 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.8 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

A53/A53M Specification for Pipe, Steel, Black and Hot-Dipped, Zinc-Coated, Welded and Seamless

[1] This practice is under the jurisdiction of ASTM Committee C16 on Thermal Insulation and is the direct responsibility of Subcommittee C16.31 on Chemical and Physical Properties.

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[2] The Uncertainty Test data have been moved to Appendix X4 because they are based on data obtained using laboratory fabricated old style test coupons. The precision and bias section, using the current practice of purchased test coupons, replaces this uncertainty data. The Uncertainty Test data is preserved (for historical purposes).

[3] 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.

A105/A105M Specification for Carbon Steel Forgings for Piping Applications
C168 Terminology Relating to Thermal Insulation
C518 Test Method for Steady-State Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus
C665 Specification for Mineral-Fiber Blanket Thermal Insulation for Light Frame Construction and Manufactured Housing
C692 Test Method for Evaluating the Influence of Thermal Insulations on External Stress Corrosion Cracking Tendency of Austenitic Stainless Steel
C739 Specification for Cellulosic Fiber Loose-Fill Thermal Insulation
C795 Specification for Thermal Insulation for Use in Contact with Austenitic Stainless Steel
C871 Test Methods for Chemical Analysis of Thermal Insulation Materials for Leachable Chloride, Fluoride, Silicate, and Sodium Ions
C1696 Guide for Industrial Thermal Insulation Systems
D609 Practice for Preparation of Cold-Rolled Steel Panels for Testing Paint, Varnish, Conversion Coatings, and Related Coating Products
G1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens
G16 Guide for Applying Statistics to Analysis of Corrosion Data
G31 Guide for Laboratory Immersion Corrosion Testing of Metals
G46 Guide for Examination and Evaluation of Pitting Corrosion

3. Terminology

3.1 *Definitions:* Refer to Terminology C168 for definitions relating to insulation.

4. Summary of Practice

4.1 The practice uses controlled amounts of test solutions delivered drip wise onto a defined area of small flat coupons of selected test metals for the purpose of producing, comparing, and measuring the corrosion that occurs on the metals due to the exposure.

4.2 The test is conducted at elevated temperatures, greatly accelerating the corrosion in comparison with corrosion at room temperature. The heat makes the solution evaporate quickly, allowing an air (oxygen) interface and making thousands of wet-dry-wet cycles possible in a short time.

4.3 Quantitative measurements of corrosion are determined from the weight change (loss) due to the corrosion of the tested coupons. Reference tests prepared with known concentrations of solutions that are conducive to the corrosion of the tested metal are compared with water solutions containing ions extracted from insulation samples. Calculations of MLCR in mils-per-year (MPY) made using the methods of Practice G1 are recorded as the quantitative measurement. The measurements are used to determine compliance with the applicable ASTM material specifications on a pass/fail basis when compared to the corrosion reference solutions that were tested at the same time as the insulation extraction solutions. No other comparisons shall be made.

5. Significance and Use

5.1 Results from this accelerated corrosion test shall not be considered as an indicator of the useful life of the metal equipment. Many factors need consideration for applicability to specific circumstances. Refer to Guide C1696 and Practice G31 for additional information.

5.2 Corrosion associated with insulation is an important concern for insulation manufacturers, specification writers, designers, contractors, users and operators of the equipment. Some material specifications contain test methods (or reference test methods contained in other material specifications), for use in evaluating the insulation with regard to the corrosion of steel, copper, and aluminum. In some cases these tests are not applicable or effective and have not been evaluated for precision and bias.

5.3 A properly selected, installed, and maintained insulation system will reduce the corrosion that often occurs on an un-insulated structure. However, when the protective weather-resistant covering of an insulation system fails, the conditions for the aqueous environment necessary for corrosion under insulation (CUI) often develop. It is possible the insulation contains, collects, or concentrates corrosive agents, or a combination thereof, often found in industrial and coastal environments. If water is not present, these electrolytes cannot migrate to the metal surface. The electrochemical reaction resulting in the aqueous corrosion of metal surfaces cannot take place in the absence of water and electrolytes. Additional environmental factors contributing to increased corrosion rates are oxygen, and elevated-temperature (near boiling point).

5.4 Chlorides and other corrosive ions are common to many environments. The primary corrosion preventative is to protect insulation and metal from contamination and moisture. Insulation covers, jackets, and metal coating of various kinds are often used to prevent water infiltration and contact with the metal.

5.5 This procedure can be used to evaluate all types of thermal insulation and fireproofing materials (industrial, commercial, residential, cryogenic, fire-resistive, insulating cement) manufactured using inorganic or organic materials, faced or unfaced, for which a filtered extraction solution can be obtained.

5.6 This procedure can be used with all metal types for which a coupon can be prepared such as mild steel, stainless steel, copper, or aluminum. Other metals (copper, aluminum) will need different times, reference solutions and cleaning practices. It shall not be interpreted that the steel procedures work for everything. When procedures are developed for other metals they will be balloted for inclusion in the document.

5.7 This procedure can also be applicable to insulation accessories including jacketing, covers, adhesives, cements, and binders associated with insulation and insulation products.

5.8 Heat treatment of the insulation (as recommended by the manufacturer up to the maximum potential exposure temperature) can be used to simulate possible conditions of use.

5.9 Adhesives can be tested by first drying followed by water extraction or by applying a known quantity of the test adhesive to a test piece of insulation and then extracting.

5.10 Insulating cements can be tested by casting a slab, drying, and extracting or by using the uncured insulating cement powder for extraction.

5.11 Reference tests prepared with various concentrations of solutions that are conducive to the corrosion of the tested metal serve as comparative criteria. Solutions containing chloride, sodium hydroxide, various acids (sulfuric, hydrochloric, nitric, and citric acid), as well as "blank" tests using only de-ionized water and tap water are used.

5.12 Research can be done on insulation that has been specially formulated to inhibit corrosion in the presence of corrosive ions through modifications in basic composition or incorporation of certain chemical additives. Corrosive ions can also be added to the insulation extraction solutions to determine the effectiveness of any inhibitors present.

5.13 Protective surface treatments and coatings of different types and thickness can be applied to the metal coupons and compared using various corrosive liquids.

5.14 Several sets of tests are recommended because of the number of factors that affect corrosion. An average of the tests and the standard deviation between the test results are used on the data. Much of the corrosion literature recommends a minimum of three specimens for every test. Consult Guide G16 for additional statistical methods to apply to the corrosion data.

6. Apparatus

6.1 The test apparatus must be housed in a reasonably clean and non-dusty environment to avoid any effects of contaminants.

6.2 *Heated Temperature Controlled Flat Hot Plate (see Appendix X1)*—A 1-ft (30.5-cm) square or circular plate that has uniform temperature across the surface provides the heated environment. See Appendix X1 for construct design and sources of assembled systems. Larger plates for testing more coupons are not excluded.

6.3 *Peristaltic Pump (see Appendix X1)*—A multi-channel peristaltic pump with individual cassettes and silicone tubes is recommended to supply 250 (±10) mL/day to each specimen. Pump rates must be well controlled.

6.4 *Silicone Rubber Tubing (see Appendix X1),* to deliver fluid to the test coupons.

6.5 *Miniature Barbed Fitting (see Appendix X1),* for connections of tubing (1⁄16 by 1⁄16 in.)(0.16 by 0.16 cm).

6.6 *Band Saw.*

6.7 *Balance,* capable of 0.0001 (±0.0002) g mass determination.

6.8 *Wet-Grinding Belt Grinder/Sander,* with used 80-grit (a belt previously used to make Test Method C692 stainless steel coupons is acceptable) or new 120-grit wet belt.

6.9 *Drying Oven.*

6.10 *Bottles,* plastic 1 L or equivalent, to individually supply each test specimen with test liquid.

6.11 *Nominal 1-in. Pipe Size – PVC Class 200 Irrigation Pipe (Thin Wall),* 15⁄16-in. (3.33 cm) OD; 13⁄16-in. (3.02 cm) ID by 1.25-in. (3.18 cm) lengths.

6.12 *High Temperature Grease or Oil,* for use as heat transfer medium.

6.13 *Rubber O-Ring,* 1¼-in. (3.18 cm) ID, 1½-in. (3.81 cm) OD, ⅛-in. (0.32 cm)thick.

6.14 *Silicone Sealant,* 100 % Silicone sealant.

6.15 *Plastic Straw,* ⅛-in. (0.32 cm) drink stirring straw ("swizzle stick") .

6.16 *Cleaning Apparatus and Solutions,* for the carbon steel coupons, Hydrochloric acid diluted 1 part to 3 parts water, razor widget, sodium bicarbonate (baking soda) solution for neutralizer, xylene, water paper or cloth towels, Wet Laid, Nonwoven Fiberglass Facing 1⁄16 in. thick – works well as a sacrificial scrubbing pad with the diluted HCL to clean and polish the coupons.

6.17 *Hand-Held Magnifier,* or 10 to 30× binocular microscope, or both.

6.18 *Filter,* 0.45 micron filter paper.

7. Reagents and Materials

7.1 *Distilled or De-Ionized Water,* containing less than 0.1 ppm chloride ions. Some de-ionized and reverse osmosis water have been found not to be pure enough. This water is used to make the test solutions and reference solutions. The "zero chloride" water test reference solution results are expected to be only slightly higher than the cleaning blank result.

7.2 *Metal Test Coupons,* meeting the composition requirements of applicable ASTM Specification for Mild Steel, Stainless Steel, Copper, or Aluminum. Mill certificates of chemical composition and mechanical properties are required.

7.2.1 Some researchers will want to maintain traceability to the metals used in other C16 corrosion procedures. Specification C739 uses cold rolled, low carbon (<0.30 %) commercial quality shim steel. Specification C665 uses cold rolled, low carbon, quarter hard, temper No. 3, strip steel. It is possible other metal grades meeting Specification A53/A53M, Specification A105/A105M, and other common ferrous steel specifications are of interest for use in the tests. If stainless steel coupons are to be used, it is recommended that they be 16-gage and prepared following the sensitization procedure described in Test Method C692 Section 9 on Test Coupons (sensitize stainless steel coupons by heating at 1200°F (649°C) in an argon (inert) or air (oxidizing) atmosphere for three hours). Galvanized steel is not suitable for test because the elevated temperatures recommended by the practice are above the recommended use temperature of galvanized metal. However, with suitable adjustments to slow the drip rate and lower the temperature of the hot plate, there are possibilities for the development of test practices.

7.2.2 Carbon Steel Coupons ; style: 0.032 Steel, Type R, Dull Matte Finish. Specs: ASTM D609-Type 1, Temper = ¼ hard, Carbon = 0.13; size = 0.032 by 2 by 3.5in. (0.8 x 51 x 89 mm)

7.2.3 It is likely that different results will be found when switching between various metal grades. The use of reference solutions of corrosive ions provides a benchmark against which the leachable ions contained in the insulation are evaluated. The reference solutions are run during every test sequence, after having previously established the range of results for the individual laboratory and the type, grade, and lot of metal.

7.3 *Chemically Pure Salts and Reagent Grade Acids* shall be used for preparation of corrosion solutions used in reference tests for plate calibration and comparison with extraction solutions.

7.4 *Chloride Reference Solutions* are prepared from a 1000 ppm (mg/L) chloride solution using 1.64 g of sodium chloride to one liter of de-ionized water. For a liter of a 1-mg/L chloride solution, mix 1 mL of 1000 ppm chloride solution to one liter. Quantity and concentration of the reference solutions are made as needed for the desired test.

8. Metal Coupon and Test Cell Preparation

8.1 Carbon steel coupons referenced in 7.2.2 are used as received from the manufacturer.

NOTE 1—The previous coupon preparation technique has been moved to Appendix X3 (History).

8.2 Permanently mark each coupon for identification. If metal stamp impressions are used to mark the coupon, do not allow the impression to deform the back face of the coupon.

8.3 Heat the coupons to drive off surface moisture and obtain a constant weight. Cool the coupons in a moisture-free environment and weigh using a precision balance to 0.1 mg. Record the weight and coupon identification.

8.4 Cut the Nominal 1-in. pipe size – PVC Class 200 Irrigation Pipe (Thin Wall) into 1.25 in. (3.175 cm) lengths. Remove the ragged edges to make smooth flat-sanded ends. Drill a ⅛-in. hole in the side of the pipe, ⅛ in. from the top end and then clean the pipe in de-ionized water and dry.

8.5 Position an O-ring approximately 0.5 in. (91.5 cm) from a smooth flat-sanded end of the PVC pipe. Put a 0.125-in.(0.32 cm) bead of silicone sealant completely around the space formed by the pipe and O-ring. Position the pipe in the center of the coupon with the hole oriented to the corner for easy access. While tightly holding the pipe down, push the O-ring into contact with the coupon, squeezing out some silicone sealant to form a continuous, watertight seal. Avoid silicone sealant on the inside of the pipe and metal. Allow the silicone to cure completely (overnight) before testing.

8.6 Cut 1-in. (2.54 cm) pieces of the plastic straw. Insert the straw into the hole in the PVC pipe so that the drip falls in the approximate center of the coupon. The barbed fitting is used to attach the straw to the peristaltic pump tube. Fig. 1 shows a completed test coupon with the components labeled. Figs. 2 and 3 show a hot plate with the coupons installed. Verify the proper setup of test coupons and solutions. It is permitted to mark the coupon and outside of the PVC cells with a position number and corresponding pump channel number.

9. Solution Preparation

9.1 *Procedure A:*

9.1.1 Many industrial insulation materials are required to meet the requirements of Specification C795 using Test Methods C692 and C871. If the material has been extracted for Test Method C871 testing, a suitable procedure is filtration of the concentrated extraction solution through a 0.45 micron filter followed by the dilution of the concentrated extraction solution with de-ionized water for use in this test. Refer to Test Method C871 for the details of the extraction. Briefly described, the procedure involves extracting duplicate ground-up samples of 20 g each in 450 g of boiling water for 30 min, adjusting the final solution weight to 500 g, and then filtering to remove the solids.

9.1.2 Combine 375 mL from each of the two extraction solutions described in 9.1.1 to provide a uniform 750-mL solution. Dilute 375 mL of the solution with 2625 mL of de-ionized water to total 3000 mL. One thousand millilitres of the resulting solution is used in a 4-day test for one metal coupon. The two extractions provide enough diluted solution

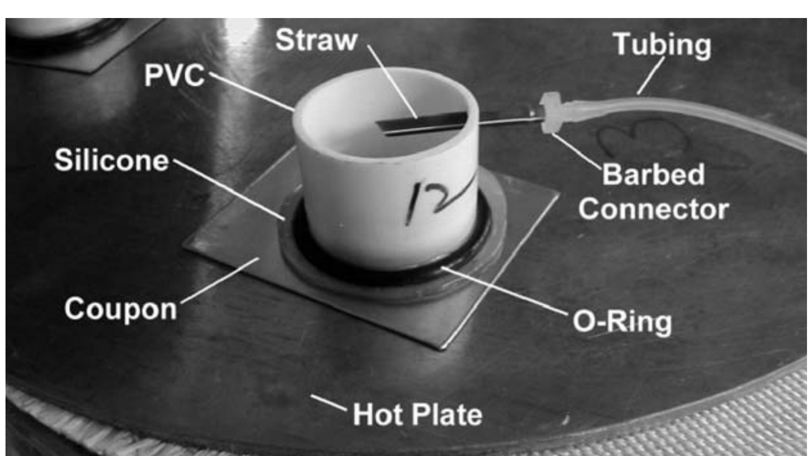

FIG. 1 Test Coupon with Components Identified

FIG. 2 Test Coupons on Hot Plate

FIG. 3 Test Cells on Hot Plate

for six coupon tests of four-day duration. The minimum recommended number of specimens per test set is three. Additional test sets are used to provide greater confidence in the results. The unused 125 mL from each of the extraction solutions are available for Test Method C871 or other chemical analysis.

9.2 *Procedure B:*

9.2.1 There are insulation materials that do not readily wick water, and cannot be made to wick by heat treatment. Some manufacturers consider it inappropriate to subject them to a severe leaching of soluble ions by Procedure A because it exposes a maximum surface area to water for extraction, which would not happen under ordinary conditions of use. An alternative extraction procedure is as follows:

9.2.2 Slice the material cross-sectionally on a band saw into 0.25-in. (0.64 cm) wide pieces. Cut enough slices so that the exposed surface area totals 2 ft^2 (1858 cm^2) . A 2-in.(5.08 cm) thick block sample would require 12 slices that are 5.11-in. (12.98 cm) long. A 1½-in. (3.81 cm) thick block sample would require 16 slices that are 4.93-in. (12.52 cm) long.

9.2.3 Record the weight of the slices.

9.2.4 Stack the slices using plastic spacers (flattened plastic stir-straws) between the slices, and secure the stack with rubber bands or monofilament fishing line.

9.2.5 Place the stack or stacks in the bottom of a suitable container. If the material floats, an appropriate means is necessary to weight the material so it remains submerged.

9.2.6 Pour in enough heated de-ionized water to cover the stack completely. If boiling water exceeds the desired extraction temperature, the manufacturer needs to specify the water temperature.

9.2.7 Agitate the contents 3 times over a 15-min period. After 15 min, filter the water though a Whatman number 41 filter or equivalent. Rinse the container and slices with de-ionized water. Record the total volume of water obtained from the extraction. Filter the extraction solution through a 0.45 micron filter.

9.2.8 Adjust the final volume to 3000 mL to test three coupons for four days.

9.3 *Reference Solutions:*

9.3.1 The use of reference tests to compare the measured corrosion resulting from the insulation solutions to that of known corrosive solutions is mandatory for the test and allows for a degree of calibration of the practice. The number of test coupons for each solution is three. Conduct the tests on the same plate at the same time as the insulation solutions.

9.3.2 The reference solutions for mild steel include de-ionized water and various solutions of chloride ranging from 1 to 5 mg/L and ideally bracket the corrosion found for the insulation coupons. Solutions that are more corrosive than 5 mg/L chloride reference solution are better tested using reduced exposure times. The reference solutions, concentrations and test times for aluminum and copper coupons include de-ionized water and various ionic solutions including chloride and sodium hydroxide, but these procedures have not been developed.

10. Test Procedure

10.1 *Test Plate Conditions:*

10.1.1 Start the heated plate previously tested and regulated to operate at 230°F (± 10°F) (100°C ±6°C) with water dripping into the test cells. The hot plate shall be maintained at this temperature throughout the test. It is permitted to start the test solutions dripping with the plate up to 250°F to help prevent the cells from overfilling. The temperature shall then be reduced to the operating range within 1 h. It is permitted to temporarily stop the peristaltic pump from dripping the solutions into the cells when cells are overfilling or an out of range temperature condition develops. Start the pump when the correct conditions are re-established. Add the stoppage time to the end of the test if necessary. Variables influencing temperature control are: the individual heated plate, digital controller (when used), thermocouple position, top copper sandwich plate (when used), and the insulation covering the thermocouple and coupons (when used). When any changes are made it is necessary to re-establish the temperature control of the test set-up. Temperature stability is improved by using thermal insulation on top of the coupons and between the cell tubes.

NOTE 2—Glass fiber felt, 0.5-in (1.5 cm) thick with an aluminum foil barrier to prevent heat transfer fluid uptake, and also EPDM-based elastomeric foam insulation have been successfully used for temperature control.

10.1.2 It is useful to test the evaporation rate of each coupon, especially on newly constructed plates, to verify that the coupons are being heated evenly. Start the peristaltic pump with the feed tubes in de-ionized water and allow the temperature controller to stabilize. Turn off the peristaltic pump and quickly fill all the test coupon cells with 1 mL of de-ionized water using an automatic pipette. Determine the time it takes for the first cell to evaporate the water (expect 2 to 3 min) and verify that the other cells dry within 45 s of the first. When necessary, reposition or otherwise adjust the coupons.

10.1.3 New plates are evaluated by performing a number of tests using only reference solutions to determine the range of the results for each solution. A Frequency Histogram similar to the one shown in Appendix X4 is developed for the individual lab, test equipment, and metal used in the test. Guide G16 is helpful in analyzing the data.

10.1.4 A small fan used to circulate the air above the test apparatus is recommended to help the evaporation process by moving the air saturated with water.

10.2 *General Procedure:*

10.2.1 Place each coupon with the attached PVC tube on the flat plate using sufficient high temperature grease between the coupon and the plate to maintain good contact (no air space). Place temperature stability insulation on top of the coupons.

10.2.2 Fill the liquid reservoirs for the peristaltic pump with the test and reference solutions and attach the individual feed tubes to the barbs in the plastic stir-straws. Record the coupon identification and solution information.

10.2.3 Start the peristaltic pump previously calibrated to deliver 250 mL/day to each sample.

10.2.4 Monitor the reservoir bottles daily to ascertain that the delivery to each sample is 250 ± 10 mL/day.

10.2.5 The time for carbon steel is equal to 96 ± 2 h to deliver the full 1000 mL of test solution per cell. No deviation is allowed other than for a power outage, a plugged tube during the test, or startup adjustments. It is not permitted to add additional solution to extend dry cells to 96 h.

11. Cleaning Coupons

11.1 The cleaning procedure is important to the accurate determination of the weight loss due to corrosion. The goal of any cleaning is to remove the corrosion product but minimize the loss of intact metal. The use of a weighed cleaning blank coupon, that is subjected to the same cleaning procedures but not otherwise tested, is necessary to determine of the weight of metal loss due to the specific cleaning procedures. Additional information about cleaning coupons after testing is written in Practice G1 Section 7 on Methods for Cleaning After Testing.

11.2 Remove the coupons and clean the heat transfer medium from the back of the coupon.

11.3 Remove the PVC cells leaving the O-ring and silicone intact when possible. At times the corrosion may be under the O-ring on highly corroded coupons. Carefully remove the O-ring while leaving the silicone intact under these circumstances. If there is loose rust or solids it is permitted to use a dental pick and water to remove any large pieces.

11.4 The following steps are for 2-3 coupons at a time and protective gloves and glasses shall be worn for the following steps. Put approximately 2 ml of 1:3 HCL (Dilute 125 ml of concentrated, 36.5-38%, HCl to 500 ml to make 1:3) in the coupon test area (within o-ring / silicone area circle only). Use a dental pick or similar instrument to gently scrape away and etch away the corrosion products. The technician shall pay close attention to the tested area and attempt to gently remove as much corrosion products as possible within any pits or groves. If necessary, the technician is permitted to scratch into the by-product to allow the HCL to react and remove the material. The HCL will become muddy during this process depending on the amount of corrosion present. The HCL stays on the tested area of the coupon for a total of 10 min even if the coupon is very clean.

11.5 Rinse under running tap water, if clean go to next step. If not, use a few more drops of HCL and work on any deep pits with the dental pick until clean. Most of the corrosion products will be removed in the previous step and only very corroded coupons with deep pits will require this extra cleaning step. Do